DEVOTED TO
TEXTILE CHEMISTRY
AND
WET-PROCESSING
BLEACHING
DYEING
PRINTING
FINISHING

MAY 2, 1949
VOLUME 38, No. 9

AMERICAN DYESTUFF REPORTER

OFFICIAL PUBLICATION
of the
PROCEEDINGS
AMERICAN ASSOCIATION
OF TEXTILE CHEMISTS
AND COLORISTS

In striving for perfect harmony your selection of color combinations will determine the degree of success of your dyeings. The best combinations are those exhibiting:

1. Practically identical migration curves

2. The same rates of exhaustion and salt sensitivity

3. Similar response to temperature

4. Compatible consumer and processing fastnesses

Here is a combination which follows this ideal pattern, but with additional advantages:

SOLOPHENYL* BROWN GL
SOLOPHENYL BROWN RL
SOLOPHENYL GREY 4 GL

Besides providing a close approach to perfect harmony in pressure dyeing (stock, beam and package), as well as in box work, these Geigy dyestuffs used together provide

SUPERIOR FASTNESS TO SUN, WASHING AND WATER, IN TAN, BROWN AND GREY SHADES AND IN THE EXTREMELY DIFFICULT TAUPES ON COTTON OR RAYON!

*Registered U. S. Patent Office

Geigy
Dyestuff Makers Since 1859

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IN GREAT BRITAIN: The Geigy Co., Ltd., National Buildings, Parsonage, Manchester

YOU CAN WIN BOTH EYE AND HAND approval with Cyanamid's Finishing Oils. They are extremely effective as softening agents for cotton, rayon and silk, and for hosiery and knitted fabrics. Grades for various textile finishing operations are available for use on the finest as well as for heavy work clothing fabrics.

YOU CAN LEVEL YOUR DYEING *and* dyeing costs with DECERESOL OT Wetting Agents because of their remarkable efficiency in achieving uniform level shades at real savings in time and expense. DECERESOL OT is the fastest wetting, penetrating and dispersing agent available.

YOU CAN SEE DOUBLE benefits — water repellency plus superior resistance to water borne stain — quickly obtained with Cyanamid's PARAMUL* 115 Water Repellent in a wide variety of fabrics . . . cottons, rayons, linens and woolens . . . for both outerwear and equipment purposes.

These Cyanamid Textile Specialties Help You LOWER COSTS AND INCREASE SALEABILITY

The Cyanamid textile specialties described briefly on this page are designed not only to lower your processing costs, but also to improve your product quality and *saleability.*

Investigate the opportunities they offer. Our textile research laboratories and technical staff are ready to co-operate fully with your staff in using these specialties to obtain the most satisfactory results.

*Our descriptive booklets, "Textile Specialties" and "*DECERESOL* Wetting Agents for Textiles" will be mailed to you promptly on request.*

*Reg. U. S. Pat. Off.

AMERICAN Cyanamid COMPANY

INDUSTRIAL CHEMICALS DIVISION, DEPT. E-5,

30 ROCKEFELLER PLAZA, NEW YORK 20, N. Y.

American Dyestuff Reporter, Vol. 38, No. 9. May 2, 1949. Published every other Monday. Copyright 1949, by Howes Publishing Co., Inc., One Madison Ave., New York 10, N. Y. Domestic subscription, $5.00; Canadian, $6.00. Foreign, $10.00. Entered as second-class matter, Nov. 6, 1919, at the New York, N. Y., Post Office, under the act of March 3, 1879.

Coprantine Colors

The way to better

WASH AND LIGHT FASTNESS
ON
VISCOSE RAYON, AND COTTON,
WOOL BLENDS AND ACETATE BLENDS
IN
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These colors . . .

are a new class of substantive dyestuffs. They give by the usual direct color procedure, dyeings which when aftertreated, combine with copper salts to form exceedingly stable complex compounds of excellent fastness to both light and washing.

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VAT DYES OF THE DOW CHEMICAL CO.

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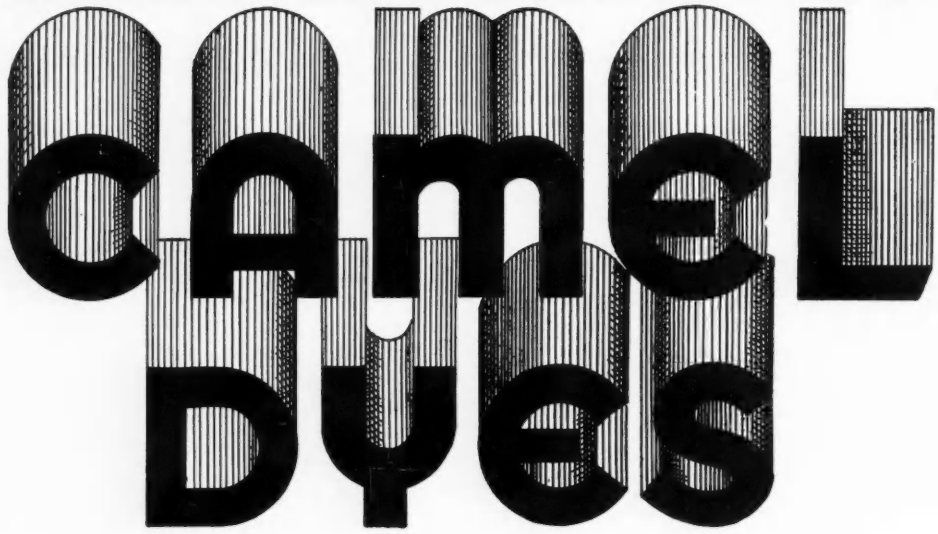

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- Aqua Ammonia
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- Tetrasodium Pyrophosphate, Anhyd.
- Sodium Metasilicate
- Sodium Sulfide
- Sodium Sulfite, Anhydrous
- Sodium Bisulfite, Anhydrous
- Oxalic Acid
- Sodium Silicate
- Aluminum Chloride, Solution
- Disodium Phosphate, Anhydrous
- Nitre Cake (Sodium Bisulfate)
- Trisodium Phosphate
- Sodium Hyposulfite (Sod. Thiosulfate)
- Aluminum Sulfate
- Lead Acetate
- Mixed Acid
- Epsom Salt
- Chrome Alum
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More Profits out of Men's Hose

These mercerized men's half hose had to be color-bright, color-fast and soft-to-the-touch to tickle the fancy of buyers. In order to obtain the fetching bold pattern and buy-appeal finish, both dyer and chemist combined their efforts in choice and application of proper dyes and finishes.

If you are experiencing difficulties with color or softness on men's half hose in your mill, find out how Amalgamated Products and Services can help you.

Amalgamated Products include Hosiery Finishes, Dyeing Assistants, Fabric Finishes and Scouring Agents. Amalgamated Field Representatives are men experienced in assisting with textile chemical problems. A modern research and production laboratory, fully equipped with facilities, is available to you for test and research. For quality fabric production and higher profits, call on Amalgamated.

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Announcement!

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amazing new developments have been taking place
in the improvement of our **AMACEL* BLACKS.**

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. . . BLACK G.S.
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As developed with Developer BON or BONASOL* these new, improved *Amacels* give the finest **developed blacks** obtainable. Perfected through the latest scientific advancements, they offer to a greater degree than ever, better fastness—simplified application—consistent shade purity. Both are suitable for box or jig . . . both are highly dispersed—**practically water soluble**—holding stain on adjacent white viscose to a minimum.

American Aniline was the first successful U. S. producer of dyes designed especially for acetate fibres, and high quality has kept *Amacels* at the top in this field.

For detailed information on *Amacel Black G.S.* and *Amacel Black IV Extra*, as well as special data regarding your own particular requirements, consult our nearest branch. A.A.P. technicians are always happy to be of service.

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*Reg. U. S. Pat. Off.

NYANZA

ANILINE & ALIZARINE COLORS

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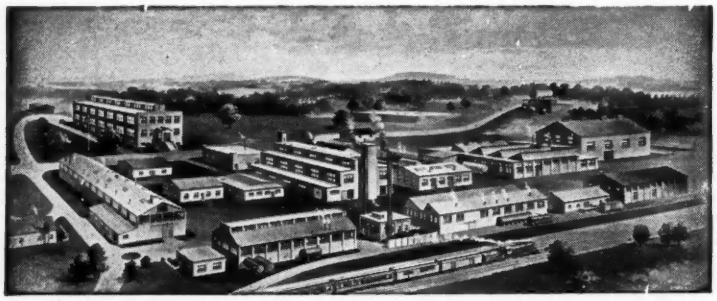
TEXTILE CHEMICALS

•

FINISHING MATERIALS

FOR

WOOL — COTTON — RAYON — ACETATE
AND MIXED FIBERS



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Ahcowet R S . . . the all-purpose
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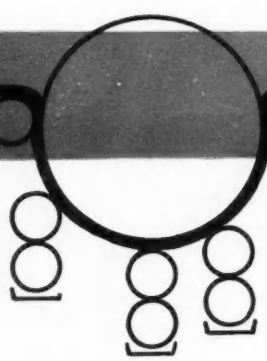
Leading printers use
Aridye Pigment Colors
because they

Cut out 3 operations

One of the outstanding advantages of Aridye® Pigment Colors is that they require no after-treatment to develop the final shade.

This means that for single-color work and multi-color patterns which can be printed with Aridye Pigment Colors alone, you can eliminate all ageing or steaming and soaping operations and cut out one drying process. After printing, the fabric is merely dried and then heat treated on an enclosed tenter or other suitable equipment.

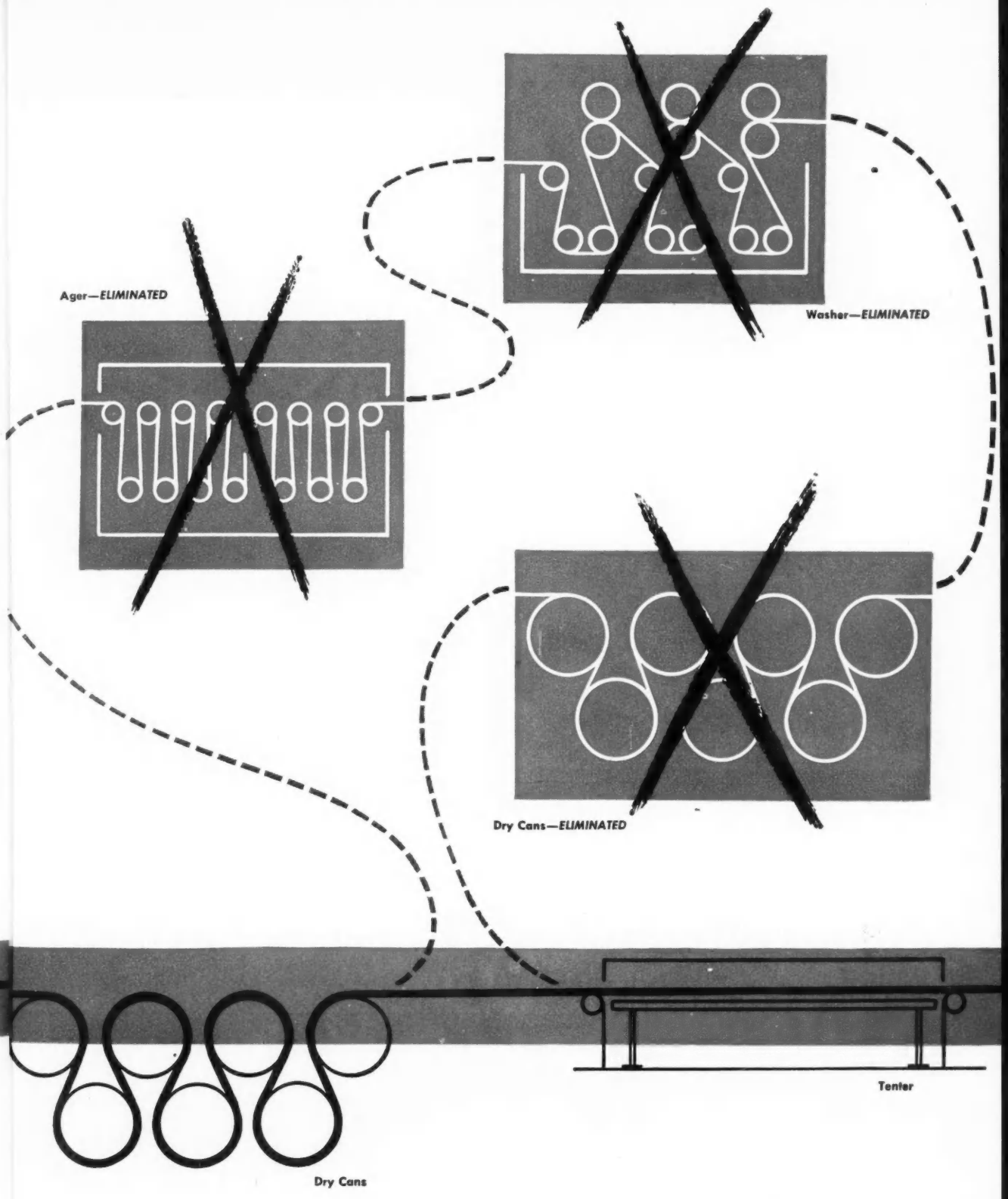
The savings in steam, chemicals and time can easily be calculated. In addition, the troubles frequently encountered in ageing are entirely avoided. Cutting out three operations speeds up production and allows quicker deliveries. An Interchemical technical representative will be glad to discuss with you in detail the many ways in which Aridye Pigment Colors can be of benefit to *you*.



Printing Machine

CHECK THESE OTHER ADVANTAGES:

Colors as printed are final in shade
Sharpness of mark
Uniformity of shade
Top fastness to sunlight
Fastness to washing
Bright colors
Ease of application
No impairment of hand
Can be used in combination with ordinary dyes



Interchemical Corporation

Textile Colors Division

FAIRLAWN, N. J. · ROCK HILL, S. C.
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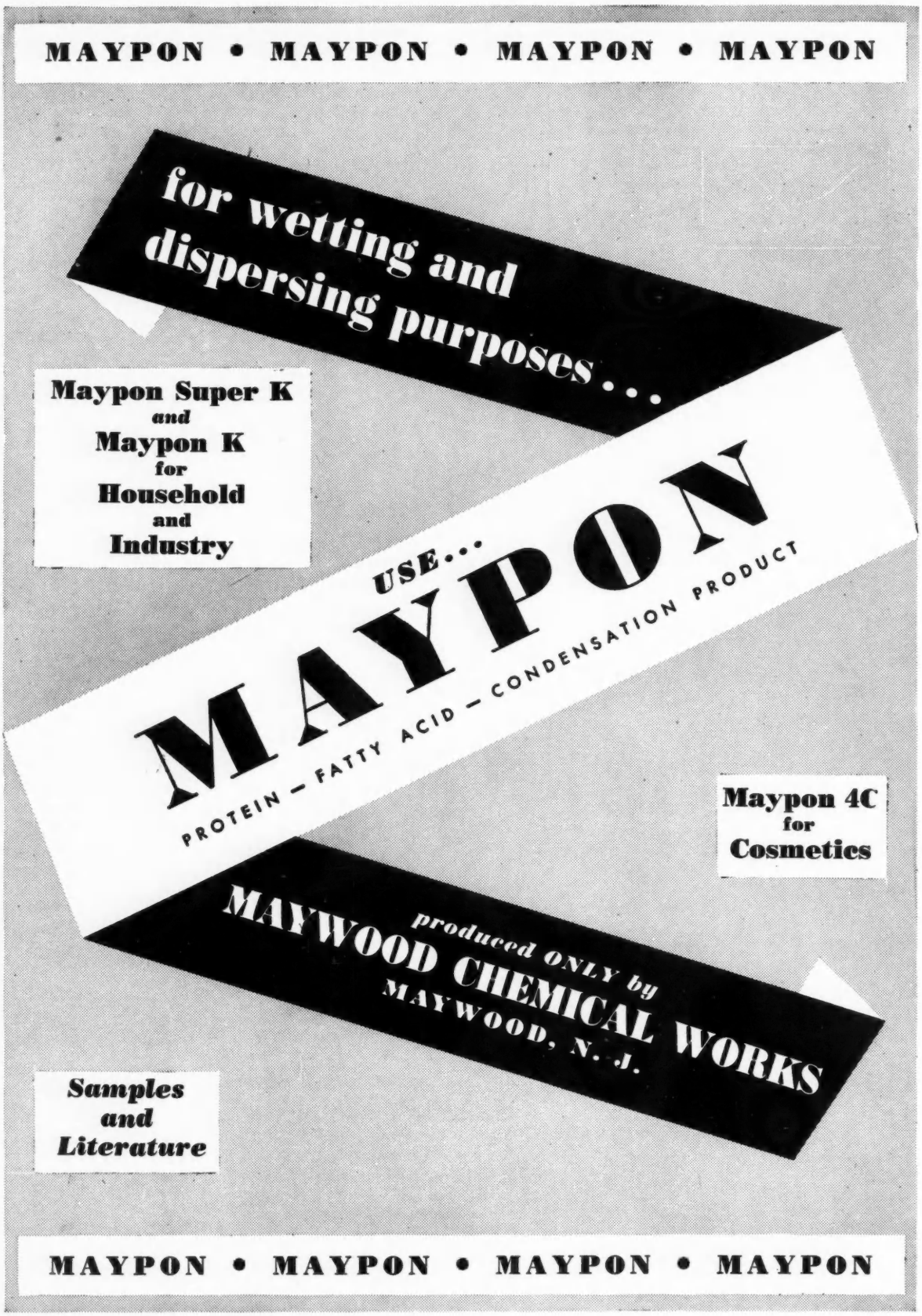
Aridye
Pigment colors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You Get Commercial Chemicals with Laboratory Purity . . .

When You Order Hooker Sulfides

With a maximum iron content of only 8PPM in Hooker Sodium Sulfide and only 5PPM in the Sodium Sulfhydrate you can understand why we are proud of the purity of these Hooker Sulfides. This minute amount of iron is matched by an especially low amount of other impurities in these Hooker products.

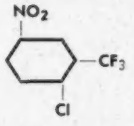
Packaged in the form of small flakes, both products are quickly and completely soluble in water. The clear solution can be used without settling or decanting. For smooth reactions—for good yields without troublesome side reactions from impurities, your safest course is to use Hooker Sulfides.

Advanced manufacturing techniques, carefully controlled operations, carefully selected raw materials account for Hooker's ability to produce these pure sulfides on a commercial basis.

As an added convenience for those who use sodium sulfhydrate in large quantities this chemical is also available in liquid form in tank car quantities. Liquid sodium sulfhydrate is shipped in concentrations of approximately 37% or 45%. Specifications will be furnished upon request.

Technical Data Sheets and samples will be sent to qualified personnel making requests on their business letterheads.

HOOKER RESEARCH PRESENTS
2-CHLORO-5-NITROBENZOTRIFLUORIDE



Synonym	2-chloro-5-nitrotrifluoromethylbenzene
Molecular Weight	225.56
Freezing Point (l.c.p.)	16.8°C
Boiling Point	230.0°C
Refractive Index n20/D	1.5009
Specific Gravity, 25/15.5°C	1.5520

Another new development of Hooker Research in benzotrifluorides is 2-chloro-5-nitrobenzotrifluoride. It is a straw colored, thin, oily liquid. Because this chemical is so new, information available on its uses is limited. Literature does indicate however, its use as an intermediate in the preparation of dyestuffs.

Hooker 2-chloro-5-nitrobenzotrifluoride is at present available in pilot plant quantities.

Those who are interested in investigating the potentials of 2-chloro-5-nitrobenzotrifluoride may obtain samples by writing on their business letterheads. Technical Data sheets giving more complete physical and chemical information are also available.

DESCRIPTION	ANALYSIS	USES
SODIUM SULFIDE Na_2S Mol. Wt. .... 78.1 M. P. ..... 100°C Light buff colored solid in flake form. Rapidly soluble in water; slightly soluble in alcohol; insoluble in ether.	Na_2S ..... 60 to 62% NaCl ..... 1.5% Max. Other Na Salts ..... 2.0% Max. Fe ..... 8 ppm Max. Cu, Ni, Cr, Mn, Pb ..... 1 ppm Max. Water of crystallization .. 36.5 to 34.5%	In unhairing hides and wool pulling; desulfurizing viscose rayon; in manufacture of dyestuffs, chemical intermediates, paper pulp, soap and rubber, as an ingredient of dye liquor for textile dyeing; boiling out linen; ore flotation and metal refining. SHIPPING CONTAINERS Steel drums ..... 90 and 350 lbs. net
SODIUM SULFHYDRATE NaSH (sodium hydrosulfide) Mol. Wt. .... 56.1 M. P. ..... 55°C Light lemon colored solid in flake form. Completely and rapidly soluble in water, alcohol and ether.	NaSH ..... 70 to 72% Na_2S ..... 2.5% Max. NaCl ..... 0.8% Max. Na_2SO_3 and $NaHCO_3$ ..... 0.4% Max. Fe ..... 5 ppm Max. Cu, Ni, Cr, Mn, Pb ..... 1 ppm Max. Water of crystallization ..... 28 to 26%	In unhairing hides, in desulfurizing viscose rayon; in preparation of dyestuffs and other organic chemicals such as thioamides, thiourea, thioglycolic acid, thio- and dithiobenzoic acids, sodium thiosulfate. SHIPPING CONTAINERS Flake: Lacquer-lined steel drums . .90; 350 lbs. net Liquid: Tank Cars .....

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℞ *for "ailing" woolen systems–*

PROXOL WOOL OIL

Proxol Wool Oil is "just what the doctor ordered" for woolen systems. When atomized and sprayed on the scoured wool, Proxol assures vibrant health for your yarn and finished goods.

℞
Here is the formula for this re-vitalizing tonic:
1. Self-emulsifying.
2. Small emulsion particle size.
3. Anti-static.
4. Non-corrosive.
5. Reduced fly or waste.
6. Quick spreading properties.
7. Excellent carding characteristics.
8. Increased spinning efficiency.
9. Uniform yarn and cloth.
10. Excellent aging characteristics— no odor or discoloration.
11. Scours easily, even after aging in the cloth.
12. Minimum residual oil content.

Your woolen system will act and feel "right on top of the world" when you prescribe Proxol. *You* will feel the same way when you observe the trouble-free operation and the quality yarn and cloth produced with Proxol.

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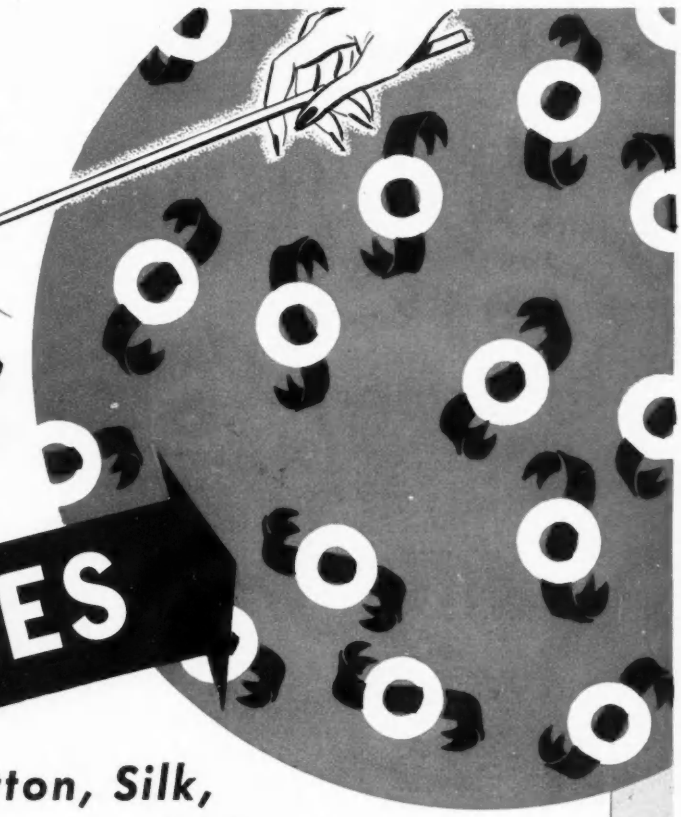
Makers of Olate Flakes the universal low titer textile soap

KELTEX works wonders WITH WHITES



On Cotton, Silk, Rayon and Wool

● In white discharge printing, you'll find that when you use Keltex as your print paste thickener you get exact and uniform paste viscosity . . . maximum penetration . . . sharp, clean-edged line . . . true, even whites.

Keltex is the modern algin thickening agent, unsurpassed for its high water-holding properties. With Keltex you have assurance of consistent uniformity, because Keltex is a definite chemical compound processed to rigid physical and chemical specifications.

Easy and economical to use, Keltex is readily soluble in cold water—requires no cooking or special treatment. Because it is entirely free of grit or cellulose, no straining or filtering is needed to insure against damage to rollers and screens. In addition, Keltex is easily washed out after printing to give your goods a soft and pliable hand.

Our Technical Service Department is prepared to cooperate with you fully in adapting Keltex to your particular requirements. Write to our nearest regional office.

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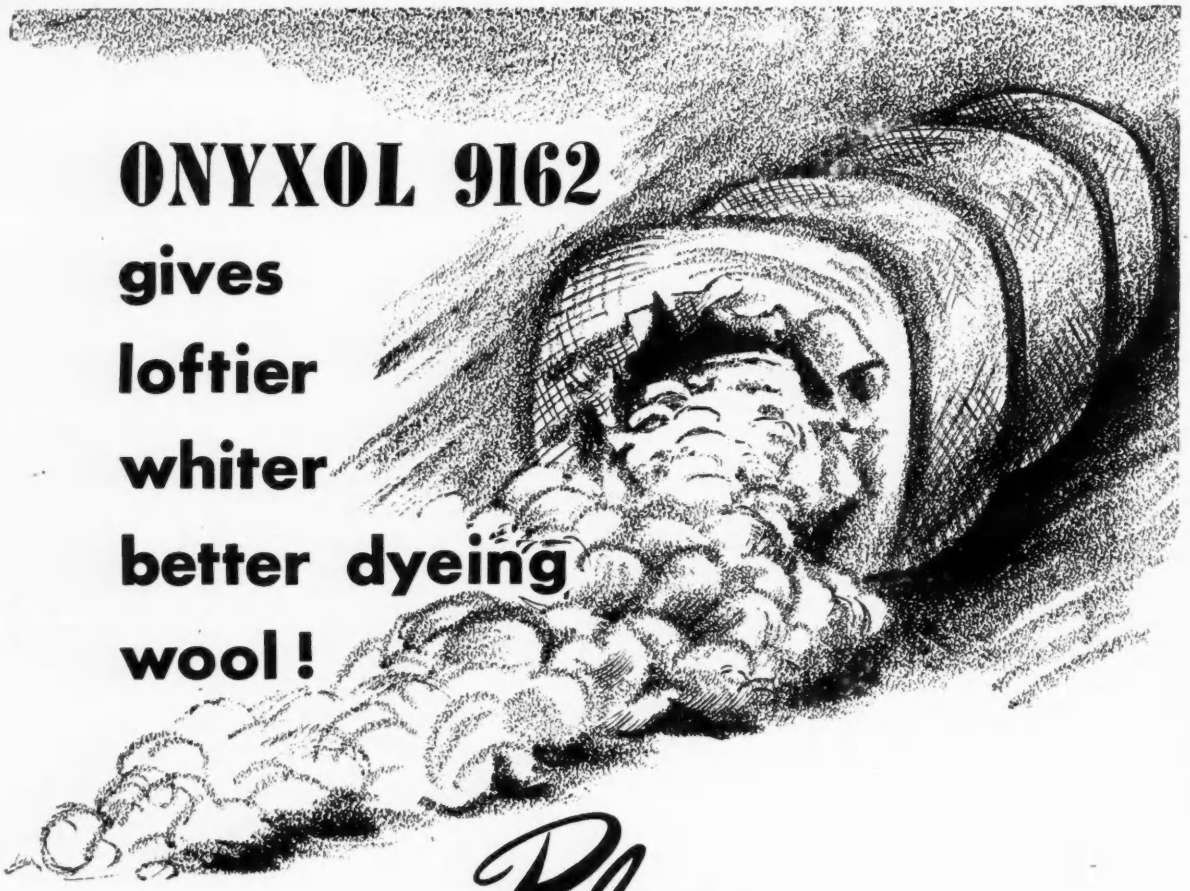

ONYXOL 9162

gives loftier whiter better dyeing wool!

Plus... faster, smoother scouring, at a cost comparable to that of high grade soap, on any type of grease wool, as well as skeins or piece goods.

These are the results indicated by: (1) A series of comparative runs with soap and with Onyxol 9162 made at the Lowell Textile Institute last Fall; (2) A subsequent mill run on 53,000 lbs. of wool, scoured with this new synthetic detergent.

You should study the complete data on these two tests embodied in Onyx Technical Report No. 19T, in order that you may reach your own conclusions as to the advisability of testing Onyxol 9162 in your own scouring train.

Write for your copy now. Please indicate if you want more than one copy of the report.

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Announcing

A New Product for DYERS and PRINTERS

LOMAR PW the new dispersing agent developed by Jacques Wolf & Co., assures more efficient and economical processing.

• *In Dyeing*

Level shades and uniform absorption of dyestuffs by the fibres, depend largely upon an even dispersion of colors in the dyebath. The addition of a small amount of LOMAR PW in pasting up indanthrenes or acetates solves this problem.

• *In Printing*

When added to the printing paste, LOMAR gives finer particle suspension, improved flow and smoother consistency. By keeping the colors in suspension, LOMAR PW assures efficient, economical results.

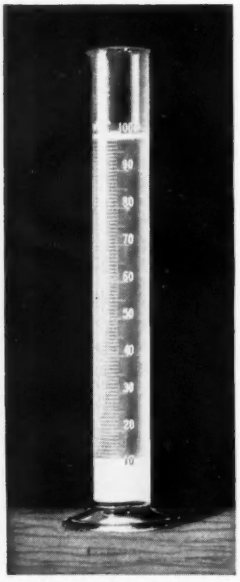
• *In Backfilling*

In backfilling, LOMAR PW is added to the heavy backfill mixture of clay and starches to give uniform suspension of the clay, good flowing properties and more uniform results on the fabric.

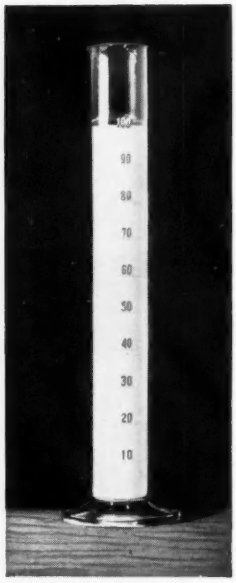
For complete information on LOMAR PW, send today for your copy of Bulletin No. 21.

TYPICAL OF THE DISPERSING ACTION OF LOMAR PW

The contents of these two graduates are identical, except for a small percentage of LOMAR PW. Note the continued even suspension and dispersion in the right hand graduate after standing ten times as long as the one at the left.



Blanc Fixe after 30 minutes — no dispersing agent



Blanc Fixe after 5 hours — plus LOMAR PW

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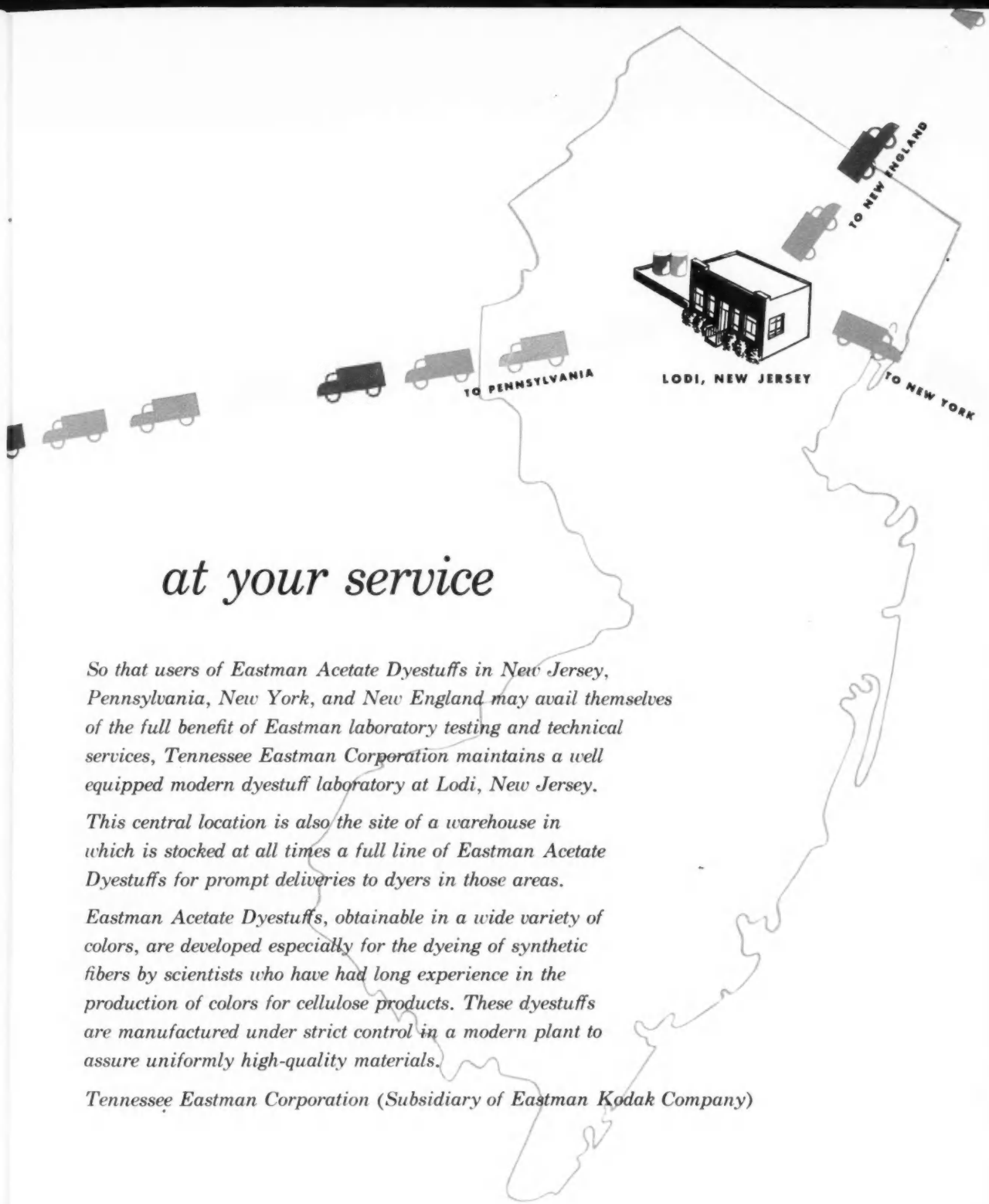
Plants: Passaic, Carlstadt, N. J.; Los Angeles, California

Warehouses: Providence, R. I.; Philadelphia, Pa.; Utica, N. Y.; Chicago, Ill.; Greenville, S. C.; Chattanooga, Knoxville, Tenn.

WOLF BRAND PRODUCTS

JACQUES WOLF & CO. *Chemicals* PASSAIC, N. J.



at your service

So that users of Eastman Acetate Dyestuffs in New Jersey, Pennsylvania, New York, and New England may avail themselves of the full benefit of Eastman laboratory testing and technical services, Tennessee Eastman Corporation maintains a well equipped modern dyestuff laboratory at Lodi, New Jersey.

This central location is also the site of a warehouse in which is stocked at all times a full line of Eastman Acetate Dyestuffs for prompt deliveries to dyers in those areas.

Eastman Acetate Dyestuffs, obtainable in a wide variety of colors, are developed especially for the dyeing of synthetic fibers by scientists who have had long experience in the production of colors for cellulose products. These dyestuffs are manufactured under strict control in a modern plant to assure uniformly high-quality materials.

Tennessee Eastman Corporation (Subsidiary of Eastman Kodak Company)

Eastman Acetate Dyestuffs

• Eastman Acetate Dyestuffs are also sold through Tennessee Eastman Corporation in Kingsport, Tennessee, and in Canada through Clough Dyestuff Company Ltd., 33 St. Mathieu St., St. Laurent, Quebec.

Shown here is one view in the spotless modern plant where Swift's Animal Protein Colloid is made. Operation is continuous, automatic . . . the product is "untouched by human hands," not contaminated by foreign substances.

Investigate this Modern Process Agent:

Swift's Animal Protein Colloid*

(made from an exclusive Swift Process)

Does your manufacturing process require . . .

1. An emulsifier?
2. A flotation agent?
3. A gel structure?
4. A settling agent?
5. A protective coating?
6. A sizing agent?
7. A carrier of pigments?
8. An impregnating agent?
9. A base for pigment?
10. An oilproofing agent?
11. A flocculating agent?
12. A clarifying agent?
13. A filtering agent?
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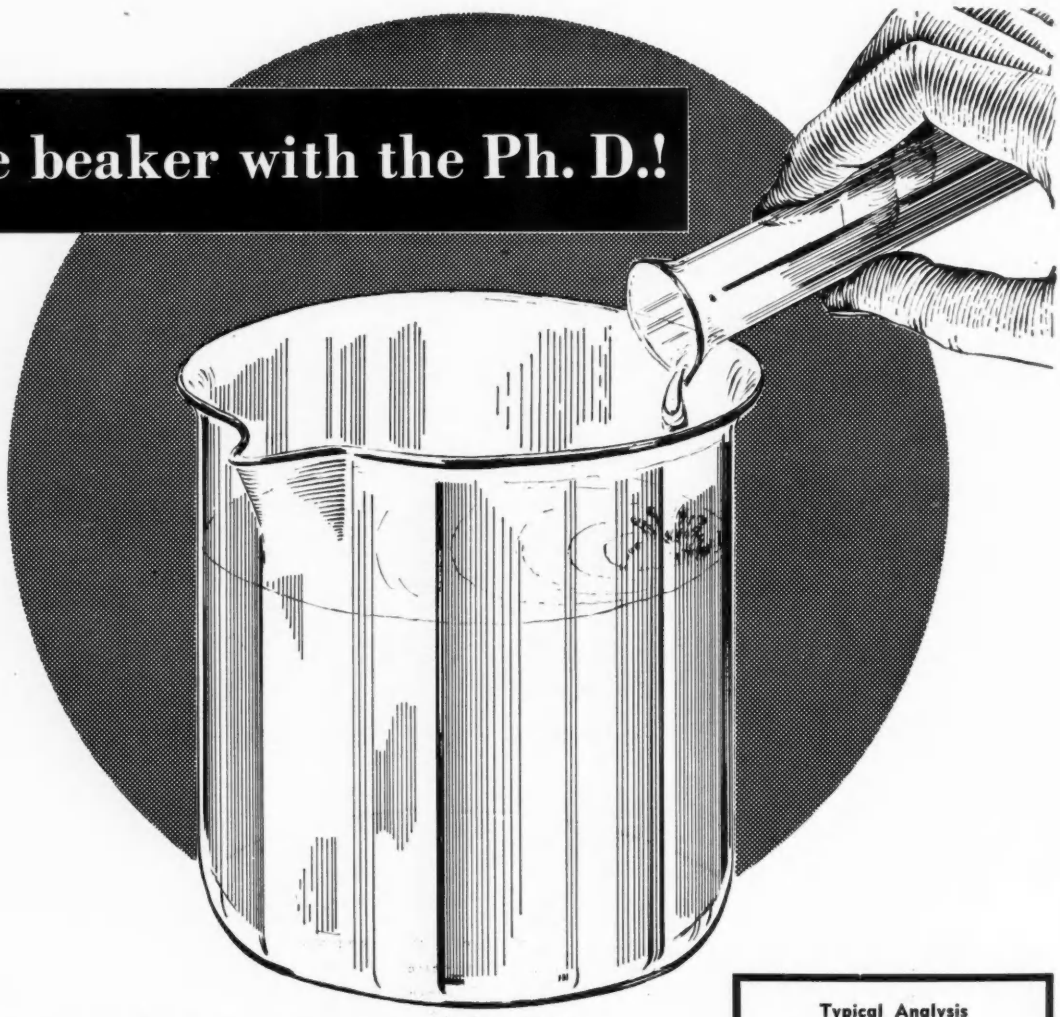

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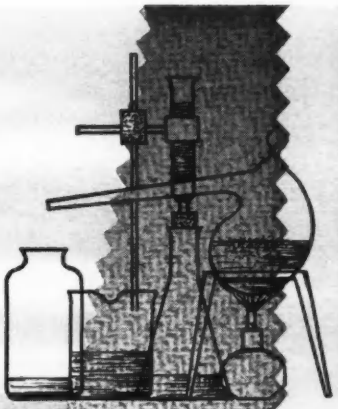

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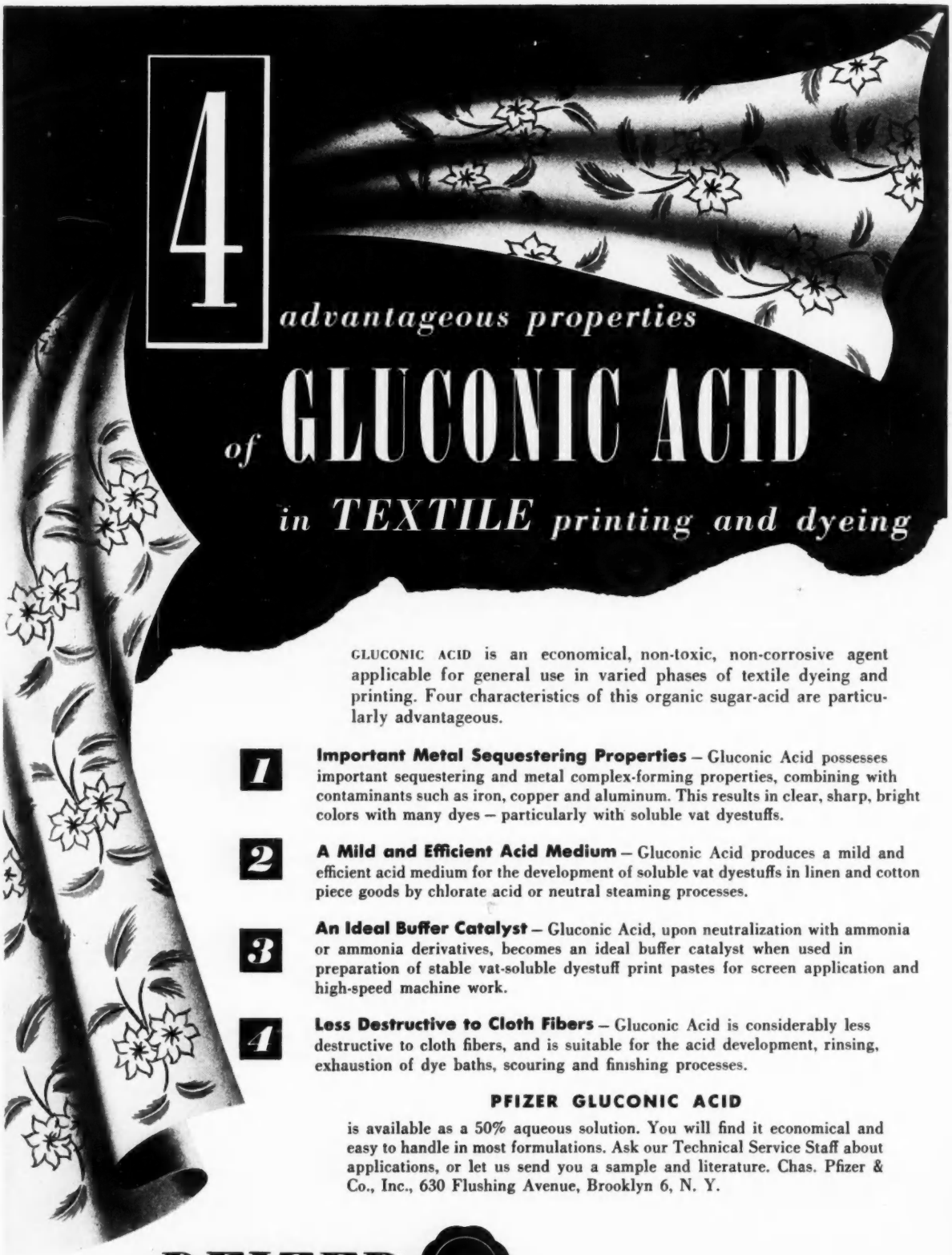

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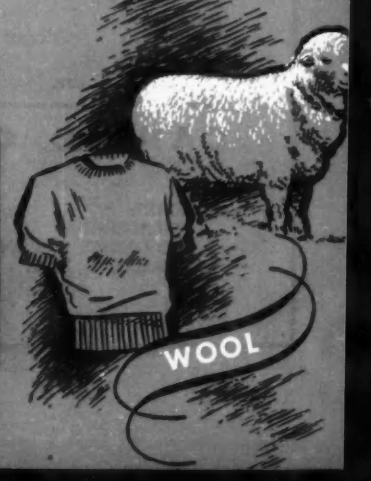

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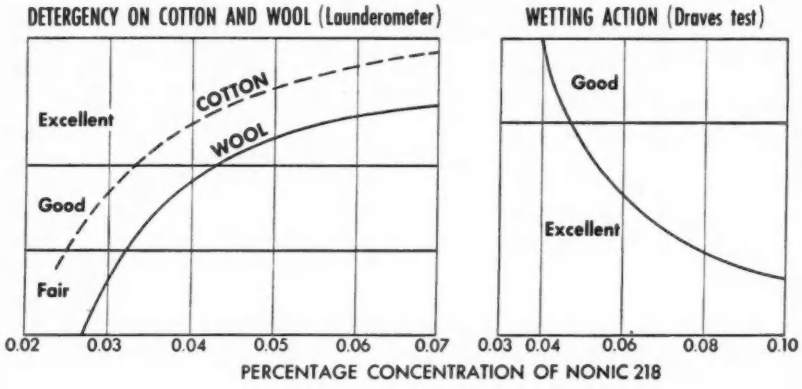

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MAY 2, 1949
VOLUME 38 NUMBER 9

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VOLUME 38 MAY 2, 1949 NUMBER 9

THE SPECTROGRAPHIC DETERMINATION OF CHLORINE IN TEXTILES

WILLIAM W. A. JOHNSON and DANIEL P. NORMAN
New England Spectrochemical Laboratories, Ipswich, Mass.

WITH the increasing use of chlorine treatments for wool and of chlorine-sensitive resins for other textiles, there is need for a rapid and accurate method for determining the chlorine content of small samples of fabric. In an earlier publication *(1)* we reported on a spectrographic method for determining chlorine and other halogens by means of a condensed spark source. The lower limit of detection of chlorine by that method was of the order of 0.5% or, on an absolute scale, 900 micrograms. This limit was not sufficiently sensitive for the purpose of determining chlorine pick-up in resin-treated textiles. It is well known, however, that exceedingly minute amounts of fluorine can be detected spectrographically as calcium fluoride *(2, 3)*. We therefore investigated the possibility of detecting chlorine in the form of a diatomic radical. The well-known Beilstein test for halogens *(4)* in organic compounds involves the burning of the halogen-containing sample on a copper wire with the consequent formation of a green copper-halide coloration in the flame. Lamb, Carlton, Hughes and Nichols showed that this test is very delicate and can be made satisfactorily quantitative *(5)*, and this test has been adopted in official specifications *(6)*. We attempted to photograph the copper chloride bands by mixing copper and chlorinated compounds and arcing them (by the technique used in determining calcium fluoride) but found that the CuCl band system consists of five extensive systems, and the energy was divided fairly equally among them so that the absolute sensitivity attainable was only a slight improvement on that obtained by the direct sparking of the sample in a CO_2 atmosphere.

Several years ago we made a study of the utilization of the CaCl band heads for the determination of Cl in a group of compounds unrelated to the present work, and, while considerable improvement over the direct sparking was obtained, the results were satisfactory only when the sample was available in certain matrices. A literature survey showed that the difficulties encountered with the CuCl spectrum would be equally troublesome in the CaCl spectrum.

Hence, it appeared necessary to find a metal halide with a fairly simple molecular spectrum, dominated by a bright band possessing a sharply defined head or other positively identifiable feature responsible for a fair fraction of the total emission. Arc runs on mixtures of aluminum powder and mixtures of halogenated organic compounds showed that aluminum chloride emission spectra at 2610 Å could be realily observed at high chlorine concentrations, but were masked by atmospheric carbon bands at low concentration. To avoid this masking, aluminum-halogen mixtures were placed in a tube of the type illustrated in Fig. 1 and the arc was operated in a vacuum. Under these conditions as little as 5 micrograms of chlorine could be detected as aluminum chloride.

The discharge voltage required to excite the AlCl band was found not to be critical so long as it exceeded 1000 volts. However, the current was found to be critical; it had to be sufficiently high to destroy an appreciable portion of a fabric sample. Chlorine values were satisfactorily reproducible at currents of 1 amp. or higher.

The following conditions were found to yield the optimum sensitivity and accuracy: One square inch of fabric was shredded with scissors and mixed with 1/10 gm. chlorine-free aluminum powder. (Most commercial aluminum powders were found to contain traces of chlorine. When a batch is found which is chlorine-free, it is reserved for this purpose.) The mixed aluminum-plus-fabric charge is placed in a cup machined from ¼" brass or copper rod and introduced into the discharge tube on a tungsten rod sealed by a graded glass seal to a standard taper joint. A pointed upper electrode is threaded to a similar tungsten support and inserted into the discharge tube so that it is 1 cm. from the surface of the sample. The exact dimensions of the assembly are of no importance so long as they are reproduced accurately on successive runs. The tube is evacuated to a pressure of 2 mm. mercury and the discharge operated by connecting the tungsten lead wires to the secondary of a suitable transformer. An exposure time of 1 min. is usually adequate to give ample light intensity. The aluminum chloride band at 2610 Å is relatively sharp, and in the vacuum discharge is not close to any other bands or lines, so that it can readily be observed with a small spectrograph of low dispersion. The accompanying photograph (Fig. 2) was made with a small prism spectrograph with a dispersion of 70 Angstroms per millimeter at 3200 Angstroms. The first spectrum on the figure is that of the pure aluminum powder. The second spectrum is that of pure aluminum chloride while the third and fourth spectra are respectively those of aluminum powder plus a wool sample and a cotton sample, each containing .02% Cl.

For a quantitative determination of chlorine it is necessary to calibrate the observed brightness of the aluminum chloride bands in terms of milligrams per square inch of fabric. For occasional determinations such calibrations can be made satisfactorily by the external stand-

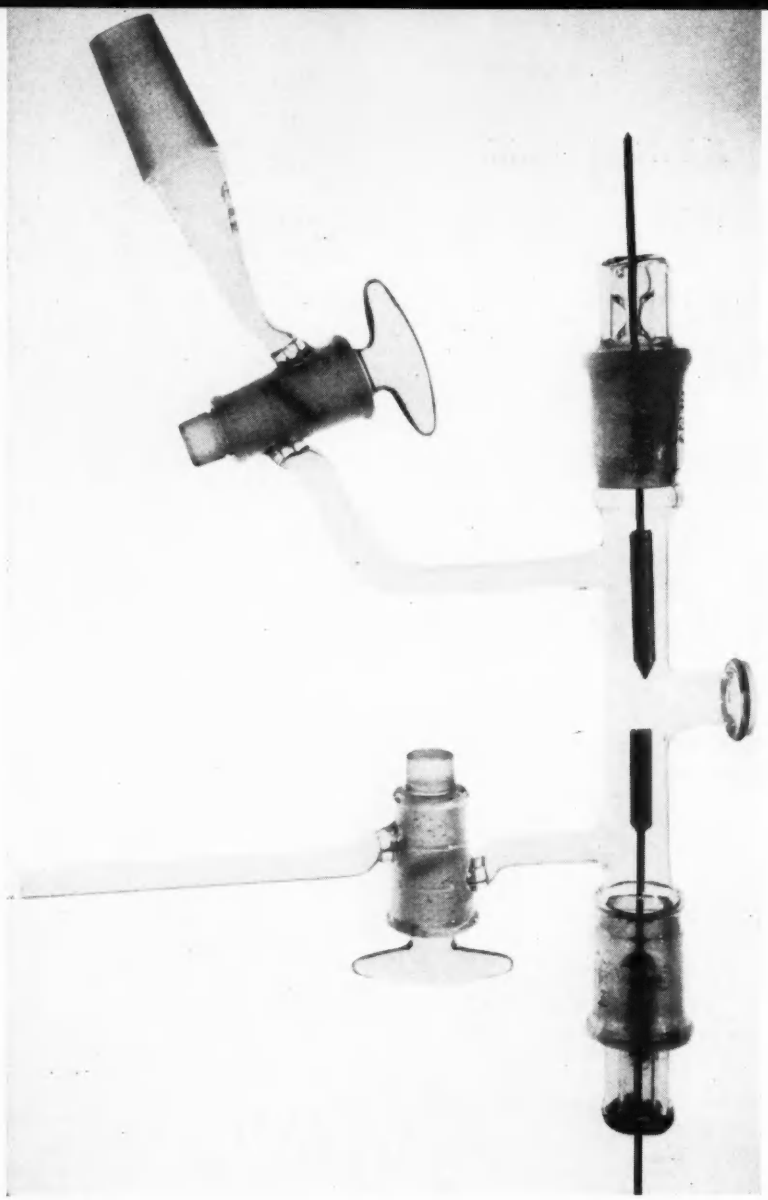

Figure 1

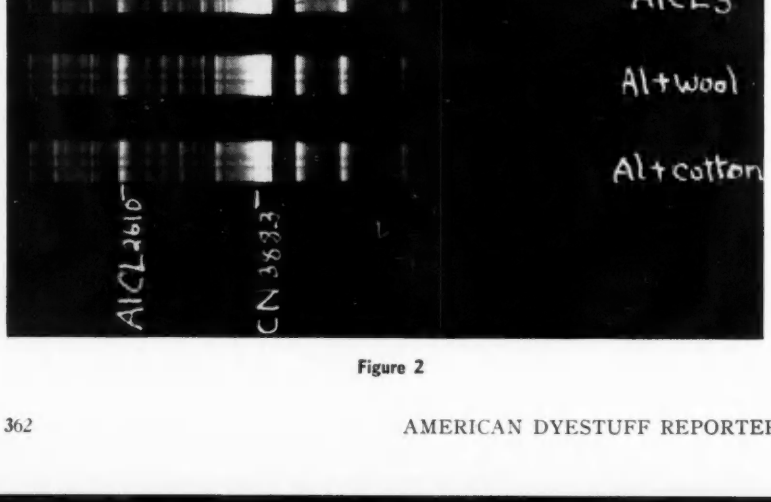

Figure 2

ard method *(7)*. For routine determinations calibration is much more satisfactorily obtained by the internal standard technique *(8)*. Both methods are described below.

1. External Standard Method

In this system the spectrum of the sample whose chlorine content is to be determined is photographed between the spectra of samples of known chlorine content. Such standards may be prepared by impregnating chlorine-free cloth with solutions containing various concentrations of a metal organic halide such as tin dichloracetate. The chlorine content of these standard samples can then be obtained directly by determining the amount of tin in the samples. Standard samples prepared by this method yielded results which were identical to those obtained with reference standards which had been chlorinated directly with hypochlorite and whose chlorine content had been determined by conventional chemical methods.

2. Internal Standard Method

In this method, a suitable reference compound is incorporated either into the aluminum powder or onto the sample so that its spectral bands are present with constant intensity in all spectra. The chief difficulty is that of incorporating a suitable material with the aluminum powder. Tin oxide can be incorporated into the Al powder by mixing, but, because of the tendency of Al to "run" or flow in grinding, this process is rather tedious if properly done.

Another technique found to be fairly satisfactory is to apply a 0.1% alcoholic solution of cadmium nitrate to the fabric sample before macerating and mixing with Al powder.

A series of fabrics of known chlorine content are spectrographed using one of these internal standards and a calibration curve is drawn relating the relative intensity of the bands of SnO or CdO and CdH to the bands of AlCl, as a function of chlorine concentration. The chlorine concentration of an unknown compound can then be determined by measuring the ratio of the brightness of its AlCl bands to the bands of the reference standard and reading the corresponding concentration of chlorine from the calibration curve. Ten samples can be measured in one hour by this system once the calibration curve has been established. The calibration curves for wool and for cotton fabrics are not identical, and separate reference standards must be prepared for each class of fabric, because the standards and the unknown textiles must absorb and retain the same amount of Internal Standard from the solution.

It is readily possible by these methods to determine variations in the chlorination treatment of fabrics or finished garments such as socks, at intervals spaced as closely as 1 cm., so that the uniformity of Cl retention as a function of garment shape or fabric weave can be readily determined.

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• PATENT DIGEST •

PAUL WENGRAF

Creaseproofing Textiles—Urea-Formaldehyde Condensed Under Specific Conditions G, 2, 04

U. S. Pat. 2,456,191
(Reichhold Chemicals, Inc., Hewett, December 14, 1948)

The specification states that urea-formaldehyde condensates have largely been described in literature and that their use in textile processing has often been recommended. U. S. Pat. 2,034,479 (1936) is especially mentioned. It comprises preparing condensates at a ratio formaldehyde: urea = 2:1. The ingredients are mixed here with acetic acid, made alkaline after condensing with a non-volatile base (NaOH) so that sodium acetate is formed in the solution. This same specification contended that the use of volatile alkalies which have to be removed later on by distillation is disadvantageous. In contrast to this reference the inventor found that the same proportion of the components (1.9-2.1 mols formaldehyde: 1 mol urea) can give a much better effect when a mixture of ammonia and sodium acetate (as buffer) is selected. Urea, formaldehyde, sodium acetate and ammonia are mixed and heated to 90°C. After two hours ammonia is removed by distillation and the batch which became gradually slightly acid, pH=5.5, was adjusted again to a value of 7.4 by adding alkali. The advantages of this process are seen in a slow formation of the resin condensate by the gradual increase of acidity, whereby small and uniform particles result which can be applied evenly and do not tend to form larger molecular aggregates. Consequently these small sized uniform particles penetrate the fiber's surface easily while particles of larger size fail to migrate through the surface and the pores.

References cited by the Patent Office, among others:

U. S. Pat. 2,263,289 (Gen. Electric): urea formaldehyde condensates are reacted in ammoniacal solution. The characteristic feature consists here of adding nitro-urea, $NH_2CO.NH.NO_2$, in alkaline solution to the resin forming components.

U. S. Pat. 2,247,764 (Nevin): adhesives are produced by combining urea, formaldehyde and cresylic acid in the presence of small amounts of ammonia, glycerine and phosphates.

U. S. Pat. 2,034,479 (MacDonough- A. D. Little): the patent quoted in the preamble of the present specification. It relates to acid condensation (pH between 3 and 5). The tendency is to avoid hydrophobic condensate formation and to obtain condensates in a hydrophilic state by establishing the proper relation between pH value, concentration of the composition and state of dissociation.

Dyeing Nylon—Direct Dyeings on Resin Impregnated Fibers C, 4, 07

U. S. Pat. 2,458,397
(Courtaulds Ltd., James H. MacGregor, January 4, 1949)

The patent specification quotes B. P. 547,844 (Courtaulds Ltd., Whittaker-Wilcock) disclosing a new method for dyeing nylon with direct dyestuffs from acid baths in the presence of an organic compound, suitable for increasing the dye affinity. Such compounds are cyanamide, melamine and other substances of this group. The present invention differs from this method in that dyeing of nylon fibers is carried out on fibers impregnated with an insoluble condensate of formaldehyde with cyanamide or derivatives thereof such as dicyandiamide, melamine, guanidine or substitution products thereof. The single components are either padded in non-condensed or pre-condensed, but still water soluble, state and cured within the fiber by subsequent heating. Not more than 20 gm. per liter of the resin-forming compounds should be used because otherwise a complex compound of dyestuff-formaldehyde cyanamide might be formed, impairing the quality of the dyeing. The method is said to be especially useful for direct dyeings, having otherwise poor affinity to nylon. Examples for this type of dyestuff are Chlorazol Fast Pink BKS (Colour Index 353) and Chlorazol Black BHS. The dyebath is kept acid like that described in B. P. 547,844. According to an example a nylon fabric is padded in a solution of diphenyl guanidine and formaldehyde, squeezed so that 70% solution is retained, dried under 100°C. and heated thereupon at 140°C. for 10 min. The thus treated fabric is dyed at 90°C. for an hour in 2% Chlorazol Fast Pink BKS and 7.5% acetic acid 80%. It was observed that the dyestuff was almost completely exhausted. The shade proved to be fuller than that on non-treated material and the hand is moreover improved, being less "soapy" than usual.

References cited by the Patent Office, among others:

U. S. Pat. 2,536,677 (same inventor): quite generally the affinity of cellulose is increased by treating the material prior to dyeing with a solution containing formaldehyde, a guanidine salt and a dicarboxylic acid (adipic acid), heating thereupon at curing temperatures.

U. S. Pat. 2,347,143 (Courtaulds): improvements in dyeing nylon with direct, acetate and acid colors are reported. The yarns are subjected in an autoclave to temperatures above 100°C.

U. S. Pat. 2,317,965 (Gen. Aniline): water resistance is imparted to direct dyeings by a pre- or aftertreatment with condensates of ethylene imine and di-isocyanate.

Other references are:

The affinity of acetate cellulose fibers (similar in their dyeing behavior to nylon) to direct, acid or sulfur dyes can be increased by incorporating acryl polymers or vinyl-maleic acid polymers (B. P. 480,297, Rhodiaceta and B. P. 459,594, Aceta). French Pat. 908,599 (Ciba) suggests likewise pre-treating material having no affinity to organic dyestuffs with a condensate of formaldehyde melamine or formaldehyde-dicyandiamide.

AN INDEX OF THE WATER-REPELLENCY OF TEXTILES FROM THE SURFACE TENSION OF AQUEOUS SOLUTIONS

H. A. SCHUYTEN, J. W. WEAVER and J. DAVID REID

*Southern Regional Research Laboratory**
New Orleans, Louisiana

METHODS of determining the water-repellency of textiles are innumerable and many reviews have appeared discussing the relative merits of the more important ones *(6, 7, 8, 11)*. Most of these methods have the utilitarian object of determining the practicality of using a particular piece of cloth for the manufacture of water-repellent clothing and are, consequently, not generally applicable to the observation of small differences in the samples tested.

During work which included a study of the relative efficiencies of various water-repellent compounds, and changes in efficiency due to method of application, laundering, and dry-cleaning, a method was desired which would be capable of critical evaluation of small differences. Since the water-repellency of the compound itself was desired, it was felt that the measurement should be as independent as possible of the type of cloth used and should measure the surface effect only. With these requirements in mind several methods were considered.

Slowinske and Pope *(11)* state that all the water-repellency tests presently used or described in the literature fall into two classes: (A) those which measure resistance to wetting of the fabric itself and, (B) those which measure resistance to penetration of water through the fabric. Tests in class A are influenced only to a minor, if not negligible, degree by fabric construction while those in class B measure a combination of water-repellency and fabric constructions and are intended to evaluate the fabrics on the basis of intended use. In either class, the basic consideration of the repellency of the fabric must begin with the nature of the repelling agent or agents. These are usually classified by use of such tests as the Spray Test *(1, 10, 12)*, Immersion Absorption *(1, 10, 12)*, Dynamic Absorption *(9, 12)*, Wetting Time *(3)*, and contact angle measurements *(4, 14)*. But each of these tests has some limitations.

The Spray Test, while being rapid and easy to manipulate, merely classifies samples into groups, without being able to distinguish between the "very good" and "excellent" samples. The values are apparently arbitrarily assigned and interpolation is invalid because the values are not numerically related. Moreover, since the falling drops have kinetic energy, fabric construction influences the results obtained. In the Immersion Absorption test it would appear that the thickness and other physical characteristics of the fabric would affect the results. Dynamic Absorption would appear to suffer from the same defects only magnified by the impact of the moving water. The Wetting Time, as well as some methods for measuring contact angles *(4)*, is ordinarily performed on yarns instead of the whole fabric, and the physical characteristics of the treated yarn may be quite different from the parent fabric. In all of these tests except those which measure contact angles, the results are not absolute, but are relative to some arbitrarily selected standard.

On the other hand when the contact angle is measured, the surface tension of the solid material is indicated although not measured directly. Unfortunately, the measurement of the contact angle of water with a fabric is not only difficult and tedious but also evaporation often increases the humidity in the immediate vicinity of the drop and changes its temperature, which may lead to errors.

Although the surface tension of the liquid is not prescribed in many of these tests, most call for "pure water" of some definite temperature, thus more or less restricting the surface tension to approximately 72 dynes/cm. On the two premises that (1) water-repellency of a pure substance is a function of its surface tension and (2) that the physical characteristics of the fabric can be measured by existing methods, there remains the need for a method of measuring the surface tension of the pure substance or of the substance after application to a fabric. Since the surface tensions of solids cannot be measured directly by any known means, an indirect method is indicated.

A method is reported here which has been devised for indicating the relative surface tensions of solids by determining the surface tension of a standard liquid which will wet the solid under selected conditions. The liquid selected was water containing various amounts of a wetting agent, prepared in such manner as to obtain a series of solutions with surface tension values ranging from 26 to 72 dynes/cm. The method will be termed the "Rolling Drop Method" of indicating water-repellency. The surface tension value of the liquid, in dynes/cm., is called the "water-repellency" index of the solid. Consider the classical equation:

$$\gamma_S = \gamma_{LS} + \gamma_L \cos \ominus$$

$$\text{or } \cos \ominus = \frac{\gamma_S - \gamma_{LS}}{\gamma_L}$$

where: γ_S = Surface tension of the solid vs. air.
γ_{LS} = Interfacial tension between the liquid and the solid.
γ_L = Surface tension of the liquid vs. air.
$\ominus$ = Angle of contact between liquid and solid.

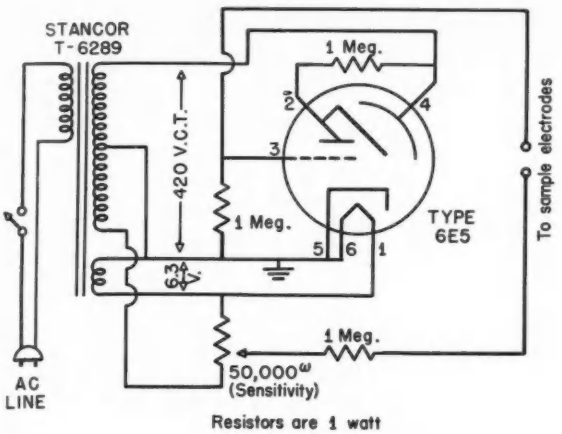
The solid will wet when $\ominus$ is less than 90°, that is, when γ_S is greater than γ_{LS}. Conversely, when γ_S is lower than γ_{LS}, the solid will not wet, since $\ominus$ is greater than 90°. For a given solid it should be possible to vary γ_L and arrive at that point where $\gamma_S = \gamma_{LS}$ and the contact angle is exactly 90°. Any value of γ_L less than this would produce wetting. Practically, however, this point is difficult to determine visually; and since time enters into almost any method of determination, the results are further obscured by evaporation of water, absoption by the cloth, and similar interfering factors.

During the preparation of this paper there was published a discussion by Antonoff *(2)* in which he makes use of his controversial equation:

$$\gamma_1 - \gamma_2 = \gamma_{12}$$

where: γ_1 = the surface tension of one of two phases in equilibrium.
γ_2 = the surface tension of the second of these two phases.
γ_{12} = the interfacial tension between the two phases.

* One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.



Resistors are 1 watt

Figure 1
Circuit Diagram of Electrical Indicator

Figure 2
Assembled Apparatus

Antonoff shows by reasoning similar to that of the present work that the point of wetting takes place when $\gamma_{12} = 0$. This would make $\gamma_S = \gamma_L$ when $\theta = 90°$, in opposition to the value just proposed for γ_S. However, if the wetting point is taken to be proportional to γ_S the argument disappears.

If the solid is inclined and the liquid allowed to drop onto the surface and roll down, the variations in contact angles become apparent by the shape of the rolling drop and the length of the "tail" which follows the drop.. As the γ_L diminishes, these "tails" become longer, depositing droplets on the surface, and finally leave a continuous trail. The γ_L necessary to produce this effect is therefore a measure of the γ_S, provided all other variables, such as temperature, volume of liquid, angle, etc., are constant. For a series of solids the various values for γ_L necessary to produce continuous wetting would be related to the values for γ_S, and therefore also related to the "water-repellency" of the solid.

Essentially, the "Rolling Drop Method" involves the determination of the highest surface tension that will wet the surface of the material being examined, as shown by the leaving of a continuous trail on the surface of the material. The visual determination of this point is difficult, especially when the solutions employed vary in surface tension by only 1 dyne/cm. By using standard solutions which are sufficiently ionic, the closing of an electrical circuit (Figure 1) has been utilized to determine the end point. Simpler circuits, could, of course, be employed, actuating any visual or audible indicator.

Experimental Results

APPARATUS.—In the preliminary experiments, the surface to which the samples are attached was constructed so that the angle and length of path might be varied. After conditions were selected, as described below, the apparatus was constructed so that the angle was fixed at 45° and the length of path was four inches. The assembled apparatus is pictured in Figure 2. The inclined panel is approximately 8 inches by 10 inches. The upper clamps were assembled from ordinary wooden clothes pin and the troughs manufactured from 20-gauge copper sheeting. These troughs are scoop-shaped, approximately 1½ inches long and ¾ inch wide at their widest part. The end of the trough which presses firmly against the sample is approximately ⅛ inch wide. These troughs are electrically bonded and connected to one terminal of the indicator circuit. The lower clamp was made from brass rod, installed under spring tension and connected to the other terminal of the indicator circuit. A trough at the lower end of the inclined plane collects waste liquid.

The electrical indicator was housed as shown in Figure 2 and consists of a tuning eye tube and its complimentary parts. This indicator tube contains a circular fluorescent screen, one segment of which is dark when the circuit is open. When the circuit is closed the entire screen fluoresces. The circuit diagram is given in Figure 1. When contact between the electrodes is established the "eye" closes. The variable resistance, shown in the drawing, was introduced to allow for increasing or decreasing the sensitivity. It was found advantageous, however, to employ the indicator at maximum sensitivity.

PREPARATION OF SOLUTIONS.—Solutions were prepared (1) with surface tensions from 26 to 60 dynes/cm. from commercially available dihexylsodiumsulfosuccinate (Aerosol MA)* in water containing 0.25% sodium chloride, and (2) with surface tensions from 60 to 70 dynes/cm. from n-butyl alcohol in water containing the same amount of sodium chloride. Any reasonably pure water may be used since the surface tensions are measured directly.

The solutions were approximated by use of concentration-surface tension curves experimentally derived, and brought to the proper surface tension by trial and error, using a duNoüy tensiometer and correcting readings according to the tables derived by Harkins and Jordan *(5)*. The measured surface tensions were accurate within 0.1 dyne/cm.

Approximate surface tensions associated with the two mixtures used in this investigation are tabulated below.

For Aerosol MA solutions:

Percent Aerosol MA in 0.25% aqueous NaCl	*Approximate surface tension*
.015	60 dynes/cm.
.050	50
.175	40
.300	35
.475	30
.620	28
.725	27
.900	26

* The use of trade names of wetting agents throughout this paper is meant to facilitate identification and is not to be taken as an endorsement of these products over any others.

For butanol solutions:

Percent butanol by volume 0.25 aqueous NaCl	*Approximate surface tension*
0.1	69 dynes/cm.
0.2	66
0.3	64
0.4	63
0.5	62
0.7	59
1.0	56
2.0	48
4.0	38

Obviously surface tension values will vary with the origin of the water and the purity of the reagents employed.

SELECTION OF CONDITIONS.—For convenience, the ambient conditions used were those of a constant temperature-humidity room (70°F. and 65% R.H.). Although the effects of different relative humidities were not fully investigated, the small variations encountered under laboratory conditions made no difference in results obtained. The lowering of the temperature of the drops by evaporation was ignored because of the rapidity of the determination. However, it is important that the measurements be made at the temperature at which the solutions have been standardized, since surface tension of pure water changes approximately 0.15 dyne/cm. for a change of 1°C.

Cloth samples giving paths 3 to 5 inches or longer could be used with little, if any, difference in results. Shorter paths gave more variable results. Four inches was selected as the length of path between the upper and lower electrodes.

Figure 3 shows the volume of standard solutions necessary to actuate the indicator, using a standard piece of cloth, plotted against the surface tension of the solution for several angles of inclination. Samples which wet absorb the liquid so that a point is reached where the volume of solution necessary to close the circuit, reaches a minimum. At an angle of 45° the volume-surface tension curve reaches its maximum change in slope at approximately 0.5 ml. and therefore this volume and angle were chosen as the fixed conditions. Angles lower than 45° produced smaller breaks in the curve while larger angles produced little if any gain in sensitivity.

The effect of the direction of path in relation to the weave of the cloth was investigated. Water-repellent 45° twill was chosen for the experiment. The drops were allowed to roll: parallel to the warp; parallel to the filling; parallel to the twill line (i.e., at an angle of 45° to the warp or filling); perpendicular to the twill line; and parallel to the warp and filling on the reverse side of the fabric. All of these positions gave the same water-repellency index.

The choice of wetting agent is a matter of convenience provided it is inert with the particular material to be tested. Besides the dihexylsodiumsulfosuccinate already mentioned, an alkyl sulfate (Duponol WS), an N-alkylsodiumsulfate substituted oleamide (Igepon T), and n-butyl alcohol—all commercially available—were tried. Results were identical. The dihexylsodiumsulfosuccinate was chosen merely because its concentration-surface tension curve was nearer a straight line than were the curves of the other agents, and this facilitated the preparation of solutions.

PROCEDURE.—Each of the samples to be tested (preferably in one inch strips) is fastened between one of the upper clamps and the lower electrode. One-half milliliter of a standard solution is rapidly introduced dropwise from an ordinary dropper into one of the upper troughs, from which the drops roll over the fabric. If the "eye" remains open, a solution having a lower surface tension is introduced into the next trough. This is repeated until the point is reached where with one solution the "eye" remains open, while another having a surface tension of 1 dyne/cm. less, causes the "eye" to close, and remain closed. The highest surface tension that will cause the "eye" to remain closed is recorded as the water repellency index of that sample.

This procedure gives satisfactory results. In a number of determinations involving ten replicates each the coefficient of variation was approximately 1%, e.g., in a typical determination, nine samples showed an index of 35, while one sample showed 36.

Results and Discussion

Eleven samples of fabric treated with four different commercial repellents were supplied by the Philadelphia Quartermaster Depot. Table I shows the treatment given the various fabric constructions.

Figure 4 shows the results of water-repellency tests given the treated fabrics, conducted, except for the surface-tension test, by the Philadelphia Quartermaster Depot. For purposes of comparison the water-repellency indices are plotted in descending order. The spray rating puts all the samples at 100. The air-permeability test shows practically no distinction between samples. Dynamic absorption shows no relation to the other tests. Hydrostatic head and drop penetration ratings do place the 9-ounce oxfords above the other samples. It seems reasonable to believe, in view of these results, that any differences shown by these tests are mainly due to fabric construction rather than to the type of compound used or the efficiency of application.

In contrast, it will be noted from Figure 4 that surface tension values vary from 35 to 42 dynes/cm. giving a range of 7 measurable units to cover the spray rating of 100. Furthermore, additional units are available for the rating as better water-repellents become available.

TABLE I
Construction of Fabric and Water-Repellent Used

Sample No.	*Construction*	*Repellent*
1	Oxford, 9 oz.	A
2	Oxford, 9 oz.	B
3	Poplin, 6 oz.	A
4	Poplin, 6 oz.	C
5	Sateen, 9. oz.	A
6	Sateen, 9 oz.	D
7	Sateen, 9 oz.	C
8	Oxford, 6.5 oz.	C
9	Poplin, 6 oz.	B
10	Oxford, 6.5 oz.	D
11	Oxford, 6.5 oz.	A

(Concluded on Page 368)

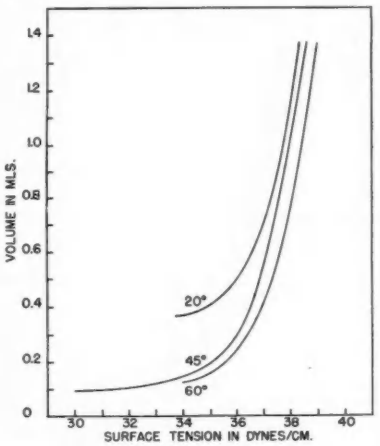


Figure 3

Volumes of Standard Solutions Necessary to Close the Electrical Circuit at Different Angles of Inclination.

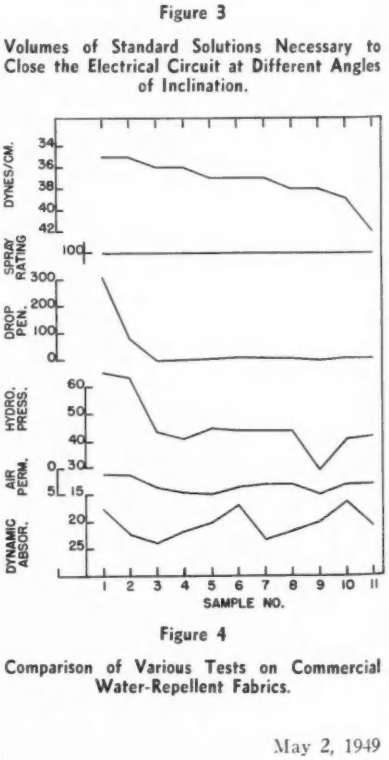


Figure 4

Comparison of Various Tests on Commercial Water-Repellent Fabrics.

COMPARATIVE EFFICIENCIES OF WETTING AGENTS ON WOOLEN CLOTH

KERMIT S. LaFLEUR
Chemist, Farnsworth Mill, Lisbon, Maine

Abstract

The comparative wool-wetting efficiencies of commercial wetting agents and allied products which lower the surface tension of water have been determined and expressed both in terms of cost and concentration. When arranged in order of performance on an equal concentration basis, dioctyl sodium sulfo succinate is most effective, and sodium rosinate is least effective of the types tested; while certain non-ionic condensates (polyethylene ethers), sodium salts of secondary alcohol sulfates, alkyl and alkyl aryl sulfonates (petroleum and coal tar derivatives), primary alcohol sulfates, and fatty acid amide and ester sulfonates show intermediate effectiveness in that order. These results supplement and appear to be in accord with previously published data.

Introduction

SEVERAL investigators *(1, 2)* have observed a correlation between the chemical structure and wetting efficiency of surface active compounds, concluding that long-chain surface active materials containing a hydrophilic end-group are in general poorer wetting agents and better detergents than branched-chain materials containing hydrophilic groups in more central structural positions. The critical factor seems to be the relative area occupied by the oriented molecule at an interface. This conclusion has been useful in supplying rough categories from which surface active agents may be selected for specific purposes, and in indicating directions for new syntheses.

The present study is an attempt to determine whether the above observation is valid for the wetting of wool, to discover where the newer non-ionic condensates and cation-active materials belong, and to supplement the comparisons based upon concentration with others based upon money-value. Since the money-value data are based upon a variable, they are of secondary importance, but they are easily correctable.

A second paper discussing the relation of detergency to the chemical structure of these same products is being prepared.

TABLE 1. Comparative wetting efficiency of competitive surface active agents on woolen cloth.

Type	Percent active ingredients	Current price per pound	Concentration (%)	Sinking time (seconds)	Lbs. per 100 gals. of solution	Cost per 100 gals. of solution	Cost index	Concentration index x 10	Performance (cost basis)	Performance (conc. basis)
Sodium salt of fatty acid mixture (25° titre)	92	.18	.10	41	.83	.15	.27	1.65	56	12
			.15	14	1.25	.23				
			.20	9	1.67	.30				
Doubly sulfated castor oil	65	.23	.09	48	.75	.17	.63	2.11	24	10
			.12	30	1.0	.23				
			.30	12	2.5	.58				
			.40	7	3.34	.77				
Sodium fatty acid ester sulfonate U. S. P. 1,881,172	51	.32	.10	43	.83	.27	1.07	2.04	14	10
			.15	28	1.25	.40				
			.25	15	2.08	.67				
			.40	10	3.34	1.07				
Sodium fatty acid amide sulfonate U. S. P. 1,192,180	16	.15	.175	44	1.46	.22	.81	1.04	19	19
			.21	34	1.75	.26				
			.35	20	2.92	.44				
			.5	12	4.16	.62				
			.65	10	5.41	.81				
Sodium oleyl alcohol sulfate	40	.30	.13	75	1.08	.33	1.00	1.60	15	13
			.18	28	1.5	.45				
			.25	19	2.08	.63				
			.40	10	3.34	1.00				
Sodium hydrocarbon sulfonate	34	.15	.08	66	.67	.10	.29	.73	52	27
			.10	43	.83	.12				
			.20	11	1.67	.25				
			.30	8	2.50	.38				
Sodium alkyl aryl polyether sulfate	30	.21¾	.075	40	.63	.14	.30	.50	50	40
			.10	23	.83	.18				
			.15	12	1.25	.27				
			.20	7	1.67	.36				
Sodium alkyl aryl amide sulfonate U. S. P. 2,277,805	35	.15	.15	59	1.25	.19	.37	1.21	41	17
			.30	13	2.08	.31				
			.40	8	3.34	.50				
Sodium alkyl benzene sulfonate U. S. P. 2,283,199	40	.15	.08	56	.67	.10	.22	.72	68	28
			.10	31	.83	.12				
			.15	14	1.25	.19				
			.20	8	1.67	.25				
Sodium alkyl naphthalene sulfonate	55	.17½	.07	50	.58	.10	.23	.88	65	23
			.08	40	.67	.12				
			.12	18	1.0	.18				
			.16	10	1.33	.23				
Sodium tetradecyl secondary alcohol sulfate	25	.33	12	96	1.0	.33	.54	.55	28	36
			.15	28	1.25	.41				
			.25	7	2.08	.69				
Sodium heptadecyl secondary alcohol sulfate	25	.33	.06	50	.5	.17	.41	.38	37	53
			.10	19	.83	.27				
			.20	6	1.67	.55				
Sodium tricarboxylic ester sulfonate	25	.24	.04	56	.33	.08	.22	.29	68	69
			.05	43	.42	.10				
			.075	20	.63	.15				
			.15	6	1.25	.30				
Dioctyl sodium sulfo succinate U. S. P. 2,028,091	25	.25	.04	34	.33	.08	.17	.20	88	100
			.05	20	.42	.11				
			.075	11	.63	.16				
			.10	8	.83	.21				
Alkyl aryl polyether alcohol	33	.16½	.05	40	.42	.07	.15	.39	100	51
			.075	19	.63	.10				
			.10	11	.83	.14				
			.15	7	1.25	.21				
Fatty acid amine condensate	100	.46½	.03	51	.25	.12	.33	.85	45	24
			.05	21	.42	.20				
			.075	12	.63	.29				
			.10	8	.83	.39				
Polyethylene ether	85	.50	.012	68	.10	.05	.16	.32	94	59
			.025	18	.21	.11				
			.05	8	.42	.21				
Dodecyldimethyl amine oxide	20	.51	.18	55	1.5	.76	1.80	.83	8	24
			.30	18	2.5	1.28				
			.40	11	3.34	1.70				
			.50	7	4.16	2.12				
Oleyldimethylamine oxide	20	.51	.25	30	2.08	1.11	3.70	1.70	4	12
			.40	21	3.34	1.70				
			.75	12	6.25	3.19				
			1.0	9	8.33	4.25				
Pine oil & emulsifier	90	.21	.15	64	1.25	.26	.38	2.02	39	10
			.20	13	1.67	.35				
			.25	7	2.08	.44				
Sodium rosinate	95	.08¾	.54	Over 2 min.		.39	.40	5.22	38	4
			.55	7	4.58	.40				
			.58	5	4.84	.42				

The least effective products in either of the two performance categories are in general not sold as wetting agents but as detergents in which capacity they excel as will be shown in a forthcoming paper on detergency comparisons.

Experimental Procedure

Circular swatches 1 inch in diameter were cut from fulled and scoured 18 oz. woolen cloth with a steel die, and wetting speed was determined at room temperature (78 ± 2°F.) by immersing a swatch in approximately 800 ml. of penetrant solution in a 1-liter beaker by means of a long-stemmed, 3-inch funnel, and keeping it submerged under a 3-inch hydrostatic head until it sank *(3)*.

Sinking times were obtained with a stop-watch, from the moment of placing a swatch on the solution surface until it finally touched the bottom of the beaker. The median of five readings was taken for each determination.

The concentrations of the individual wetting agents necessary to obtain wetting speeds between arbitrarily chosen limits of 50 to 10 seconds were experimentally determined; these concentrations were then expressed in terms of cost per 100 gallons of wetting solution, and this cost was plotted on cross-section paper against sinking times to produce wetting curves comparable on a money-value basis.

The cost per 100 gallons of a wetting solution at a concentration necessary to produce a sinking time of 10 seconds was called the room temperature cost index, and it expresses quantitatively the current money-value relationships of the penetrants tested.

Similarly, the concentration of each product necessary to produce a sinking time of 10 seconds was obtained from sinking time-concentration curves. This value, corrected to a 100% basis from the percentage of active ingredients, was called the room temperature concentration index.

The relative efficiencies of the products examined were computed from the concentration and money-value indices by assigning the value 100% to the best product in each category.

Data are shown in Table 1.

Discussion

Although several significant variables were disregarded in these tests (the influence of electrolytes *(2)*, the influence of chain length *(4, 5)*, the effect of higher temperatures*, *(6)* and pH**), a scatter diagram plotting performance against the anion-active structural types shows that good correlation exists between the two.

On the non-ionic condensate types examined, the polyethylene ethers are very effective, while the fatty acid amine condensates are only moderately so.

The two cationic-active agents included indicate that longer chain length favors greater efficiency. This conclusion would probably be qualified by additional work *(5, 6)*.

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* Cost indices decrease with temperature (e.g. sodium salt of fatty acids gave .27 at 77°F, .24 at 100°, .20 at 140°, and .16 at 200°), but at variable rates (e.g. sodium heptadecyl secondary alcohol sulfate gave .41, .39, .36 and .35; sodium alkyl benzene sulfonate, .22, .18, .14 and .13 at 77, 100, 140 and 200°F.).

** The cost indices of dioctyl sodium sulfo succinate, a sodium hydrocarbon sulfonate, and a polyethylene ether in a 4% sulfuric acid solution were .38, .26 and .19 respectively, compared to .17, .25 and .16 in water solution.

Index of Water Repellency—

(Concluded from Page 366)

Although these results indicated that the "Rolling Drop Method" was a sensitive measure of slight changes in surface water-repellency, it was recognized that the differences might not be significant. Therefore, the following series of tests was made:

Samples of commercially treated, water-repellent, 7 ounce, 45° twill were washed by modification of the colorfastness to laundering test procedure No. 3 of the National Bureau of Standards *(13)* using a laboratory shaker in place of the standard washing machine, and dry-cleaned according to the National Bureau of Standards' test for colorfastness to dry cleaning *(13)* with the same modification. The samples were treated a sufficient number of times to lower the spray rating to 70, removing, however, a sample after each treatment. It was believed that such treatment would give a reasonably slow and even break-down of the water-repellent surface. The results are shown in Figure 5. From the variation in air permeability, it is evident that there were some mechanical changes in the cloth, partially reflected by the change in hydrostatic pressure. While the spray ratings were reduced from 100 to 70, the surface tension indices varied smoothly from 36 to 58 dynes/cm. giving a range of 22 measurable units, mathematically related to one another, compared to the 4 arbitrary units employed in the spray test. Furthermore, these units are determined by means of an instrument instead of visually.

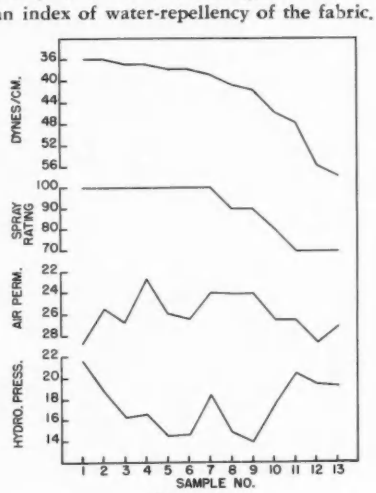

Figure 5

Comparison of Various Tests on a Wash Degraded Water-Repellent Fabric.

Conclusions

By the use of solutions whose surface tensions are known and by employing an electrical indicator, the point where fabrics become wet can be determined.

The surface tension of the solution necessary to cause this wetting is taken as an index of water-repellency of the fabric.

This system gives a range of at least 22 measurable units compared to 4 arbitrary units of the much used spray test.

Acknowledgments

Our thanks are extended to Edmund M. Buras, Jr., of this laboratory for the design and construction of the electrical indicator used in our experiments.

We are indebted to M. I. Landsberg of the Philadelphia Quartermaster Depot for the rated samples of water-repellent fabrics; to H. M. Chase of Dan River Mills, Danville, Virginia, for the sample of water-repellent twill; and to Dr. Ruby K. Worner for test data on the latter material.

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Proceedings of the
American Association of Textile Chemists and Colorists

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May 2, 1949

CALENDAR

SOUTH CENTRAL SECTION
Meeting: May 21 (Patten Hotel, Chattanooga).
WESTERN NEW ENGLAND SECTION
Meeting: May 20
Outing: June 24
NEW YORK SECTION
Meeting: May 6.
Outing: June 17.
PHILADELPHIA SECTION
Meetings: May 20, September 16, November 11 (Kugler's, Philadelphia).
Outing: June 10.
PIEDMONT SECTION
Meetings: June 24, 25, 26 (Ocean Forest Hotel, Myrtle Beach, S. C.), November 5 (Charlotte Hotel, Charlotte, N. C.)
HUDSON-MOHAWK SECTION
Meetings: May 20, September 16, November 18.
Outing: June 17.
PACIFIC COAST SECTION
Meetings: May 13 (Los Angeles); May 17 (Portland).
RHODE ISLAND SECTION
Meeting: May 20
Outing: June 24
MID-WEST SECTION
Meeting: May 7 (Hotel Schroeder, Milwaukee).
Outing: June 18 (Lake Lawn, Delavan, Wis.).
NORTHERN NEW ENGLAND SECTION
Meeting: May 20 (Andover Country Club).
COUNCIL
Meetings: June 9, Sept. 9, Dec. 9.
RESEARCH COMMITTEE
Meetings: June 9, Sept. 9, Dec. 9.
NATIONAL CONVENTIONS
1949: October 13 to 15, Chalfonte-Haddon Hall, Atlantic City.
1950: October 19 to 21, Statler, Boston.
1951: October 18 to 20, Statler, New York.

Western New England Section to Meet May 20th; Outing on June 24th

THE next meeting of the Western New England Section will be held on May 20th at the Waverly Inn in Cheshire, Connecticut. The meeting has been designated as "Ladies' Night" and wives and lady friends of members are invited to attend. There will be an exhibit composed of products of mills in the area.

Speakers for this meeting will be Dr. Jules Labarthe, Jr., a Fellow at the Mellon Institute and Dr. Martha Johnson, Manager of the Food Research and Appliance Testing Laboratory at General Electric Company in Bridgeport. They will discuss consumer problems as related to the textile industry.

The Western New England Section will hold its annual outing on June 24th. The place will be announced at a later date.

Rhode Island Section Meetings

Shown above is W. P. ter Horst, General Manager, Fiber Division, Virginia-Carolina Chemical Corp., who spoke at the February 25th meeting of the Rhode Island Section on "Vicara—New Fiber Derived from Zein". A number of samples of the fiber are also shown.

Photos by Kenneth C. Everett, Danielson, Conn.

At the March 25th meeting of the Rhode Island Section are shown Emory A. Richards, Secretary of the Section, Elliot Broadbent, Vice-Chairman of the Section, and Dr. Henry W. Grimmel, Vice-President and General Manager, Metro Dyestuff Corp. Dr. Grimmel discussed "Headaches of a Dyestuff Chemist".

RESEARCH COMMITTEE REPORTS

Presented to General Research Committee Meeting, March 25, 1949

Fastness to Light

IN our last report it was stated that the new Calibration Paper No. 1554 and its faded standards had been approved by the Committee and that distribution would be made by the National Bureau of Standards. We can now report that 150 sets of papers and standards have been sent out, and the balance of over 200 requests will be filled as rapidly as the production of the faded standards will permit.

There is nothing further to report at this time on the investigation of the GE Totalizing Photometer, which is being carried out both at the Bureau of Standards and by Atlas Electric.

The Fade-Ometer faded swatches of A.A.T.T.C., British and German light fastness standards are being circulated among committee members for critical evaluation, and Mr. Christison has been supplied with an additional quantity of the German Standards for some further exposures and comparisons, both in Fade-Ometer and sunlight. No final conclusions are yet possible, but a preliminary opinion would be that the A.A.T.C.C. standards, though not perfect, excel the European standards in better gradation between successive levels of fastness.

W. A. HOLST

* * * *

Washfastness

AT the last meeting in January we reported that the research associate at Lowell had developed a test method, which could be performed in 45 minutes, to produce results comparable with the average effect of 5 commercial launderings and that to do this it was necessary to replace the present 1 pint glass jars with tubes measuring 12 by 2 to 3 inches in diameter, and that on request we had received approval from E.C.R. to purchase several of these tubes at a cost not to exceed $100 for distribution to several laboratories together with dyed and printed test samples to learn how their tests by the new test method compared.

The 12 by 3 inch tubes were not considered completely satisfactory because of their cost about of $20 each, because of their weight of several pounds and because their use would reduce the capacity of the Launderometer to one-half its present capacity.

Further experiments indicate that a tube or jar measuring 8 inches by 3 inches can be used as a substitute for the larger tubes and this substitution would maintain the present Launderometer capacity at 20 tests, reduce the cost of the tubes to about $10 each and reduce the weight considerably. Joseph Norton of the Atlas Company has stated that he would learn from Corning Glass at what cost the metal tube could be duplicated with Pyrex glass and then be prepared to furnish both the metal tube and a glass tube. Atlas states that they prefer to furnish the 8 by 3 inch metal tube because it can also be used in the Launderometer for dyeing leather and textiles and that they are now in use for these purposes. The tube is sealed with a rubber ring and a spring clip and can be closed and opened as easily as the present pint jars. Atlas is not quite ready to furnish 10 to 12 of these new tubes for our planned co-operative laboratories study, stating that they should like to prepare single jars with several changes with the hope that these changes will effect some improvements. These single jars will be furnished Lowell for further experiments and we hope that we have these completed by the time of our next Research Committee Meeting. In the interim the search for dyed and printed fabrics to serve as standards and controls will be continued. It is planned to develop a set of 3 or 4 standards or controls to be made available for those who wish to use them.

In the work by the Synthetic Fibers Division it was disclosed that the ⅜ to ½ inch diameter rubber balls effected a greater degree of color damage than the ¼ inch diameter stainless steel balls now used. The rubber balls were discarded because they were not available during the war time period and because they stained white wool, leading to confusion in test interpretation. We are informed that the rubber balls are now available in improved quality, free of uncombined sulfur, which should eliminate the objectionable staining of white wool and Atlas has agreed to learn if synthetic rubber balls are available. Tests will be made with the rubber balls in an effort to learn if they can be used to advantage compared with the present metal balls.

CHARLES A. SEIBERT

* * * *

Fire Resistant Materials

FOLLOWING the fire resistance tests which the Committee conducted in January in conjunction with the American Cyanamid Laboratories, the data were studied critically and it was decided that this phase of our work should be repeated. Two new fabrics will be included in the new test series and a total of six testing apparatus will be used.

As previously reported, in the most recent tests the A.A.T.C.C. apparatus was much more severe than both Cyanamid apparatus, and it appeared that the A.A.T.C.C. sample holder was the reason for the increased severity. At Stamford it was found that the tests using the Cyanamid apparatus were run with a 1¾ inch frame. This deviation from the A.A.T.C.C. requirement of a 1½ inch flame was unintentional and was not detected until after the supply of test samples had been exhausted. Under the circumstance of this 1¾ inch flame, the fabric sample extended one inch into the flame. Where the 1½ inch flame was used with the other two apparatus, the fabric sample extended ¾ of an inch into the flame.

Curiously enough, the Stamford tests with a 1¾ inch flame seemed more severe than the identical apparatus at Bound Brook with a 1½ inch flame where Fabric "A" flame duration was concerned, but less severe where Fabric "B" flame duration was concerned. The need for repeating this work is evident.

The plan of approach for the new test series has been prepared very carefully by A. J. McQuade and Gerald Winston of the Philadelphia Quartermaster Corps.

A sampling plan was devised to facilitate correlation of results to be obtained on six different testing apparatus. Preliminary tests were run on the test fabrics, and the sampling plan is based upon the results of these preliminary tests. The fabrics are ready for distribution and this work is going forward. We should have some interesting progress to report at the next meeting of this body.

JAMES R. REDMOND

* * * *

Analysis of Fiber Mixtures

THE Committee is at present working on the following projects:—

(1) A study of methods for Vinyon, Vinyon N, and Orlon.

(2) A study of methods for regenerated protein fibers.

(3) A revision of the 70% sulfuric acid method for wool blends.

(4) A revision of the conc. hydrochloric acid method for silk blends.

(5) Continuing work on the 20% hydrochloric acid method for nylon.

LESLIE CHRISTISON

* * * *

Chlorine Retention

THIS committee has completed many tests on rayon fabric using five different resins and will now investigate several chlorine treatments. After the chlorine treatments have been completed the fabrics will be tested at various temperatures and for different times on the Suter tester. A tentative approach has already been agreed upon within the committee and confirming tests to determine the duplicability will be run off. There is a possibility that a report will be ready by June.

ARTHUR ETCHELLS

* * *

Dimensional Changes

Progress Report of Boston Area Sub Committee on Wool Fabric Shrinkage

IN response to the various suggestions of the Steering Committee for further investigation by the Boston Area Committee, comprehensive tests were conducted in the Pacific Mills' Laboratories. It is not possible to include the complete data, photostats, etc., within this report but the conclusions arrived at as a consequence of these studies are as follows:

A. The shrinkage in laundering of stabilized wool fabrics is slightly greater when soap is used as a detergent than when a synthetic detergent is used.

In these tests 9 samples each of 3 different stabilized fabrics were laundered 7 times at 100°F. for 30 min. with two 5-min. rinses. In one series of tests Ivory soap was employed as the detergent while another series used a synthetic. The felting shrinkage results obtained are noted—

	Felting Shrinkage in 7 Launderings	
	Warp	*Filling*
Chlorination A Soap	2.1	1.7
Chlorination B Soap	8.1	2.7
Resin	2.0	0.2
Chlorination A Synthetic..	1.3	0.4
Chlorination B Synthetic..	5.3	2.1
Resin	1.2	0.2

B. A series of tests for the evaluation of wool fabric relaxation procedures were conducted in order to determine the relative effectiveness of cold water spraying and soaking. Drying methods also were investigated in connection with this study and included pressing, oven drying and tumbler drying.

It was established that the relaxation shrinkage resulting from cold water spraying was practically identical with that obtained by soaking for 2 hours with a wetting agent. The method of drying, however, produced a significant relaxation shrinkage. The method of sponging or wetting appeared to have no important effect on degree of felting shrinkage obtained when the relaxed fabrics were subsequently laundered three times according to the tentative standard washing method, or 17 times according to CCCT191A. Tumbler drying gave significantly higher felting shrinkages (in CCCT191A series of washings) apparent after 7 launderings.

C. A series of tests designed to determine correlation between commercial launderings and the tentative standard test showed that two of the 75 min. accelerated launderings were equivalent to 15 commercial launderings in shrinkage. It was observed that short successive launderings (CCCT191A) consumed too much time to be practicable in control testing and soap pick-up was cumulative and arrested shrinkage. Two of the 75 min. accelerated tests are more strenuous but are roughly equivalent to 15 CCCT191A washings.

D. Comprehensive tests were also made in an effort to appraise the influence of water level and temperature on wool fabric shrinkage when the 75 min. tentative washing method is employed. As a consequence of these tests it was concluded that "there appears to be a critical water level at a given temperature." In the washer used (Prosperity "Junior") this level was 8". One washing at 8" at 100°F. produced the same shrinkage as two washings at 10".

"Increased temperature (100°-110°-120°) at a constant water level did not have a direct effect on felting shrinkage."

"The optimum conditions for accelerated wool laundering appear therefore, to be at a water level of 8" (Prosperity "Junior" washer. at a temperature of 100°F. One 75 min. washing at 8", 100°F. is more effective than 15 American Institute of Laundering or CCCT191A launderings."

A. I. ANDERSON

—♦ ♦—

Northern New England Section to Meet May 20th

THE next meeting of the Northern New England Section will be held on May 20th at the Andover Country Club, Andover, Massachusetts. Dinner will be served at 6:00 P.M.

The speaker will be Robert D. Robinson, Chief Chemist, Bachman Uxbridge Worsted Corporation, Uxbridge, Massachusetts. His paper is entitled "Some Practical Aspects of High Temperature Dyeing".

Dinner reservations should be made with

Edward B. Bell,
Textile Aniline & Chemical Company,
49 Blanchard Street,
Lawrence, Massachusetts.

—♦ ♦—

May 7th Meeting, Mid-West Section

JACK EPELBERG of Cluett, Peabody & Company will present a paper on chlorination treatments at the afternoon session of the meeting of the Mid-West Section to be held on May 7th at the Hotel Schroeder in Milwaukee. Dinner will be served at 7:00 P.M. and the speaker of the evening will be J. Robert Bonnar, chairman of the A.A.T.C.C. General Research Committee, who will discuss the "Inner Workings of A.A.T.C.C. Research".

—♦ ♦—

Rhode Island Section to Meet May 20th; Outing on June 24th

THE May meeting of the Rhode Island Section will be held on the 20th instead of the 27th as originally scheduled. It will be a dinner meeting at Johnson's and Fred Grant is in charge of dinner arrangements.

Speakers for this meeting will be:

Asa Francis Fisk, Stowe-Woodward, Inc., "Current Trends in Operating Rubber-Covered Rolls in Textile Finishing."

H. F. Creegan, Rodney Hunt Machine Co., "Modern Trends in Dyeing and Finishing Machinery."

The Rhode Island Section has scheduled its outing for June 24th.

Hosiery Group Meeting—

LABORATORY EVALUATION OF SIZING MATERIALS FOR NYLON HOSIERY YARNS*

HERMAN B. GOLDSTEIN

Director of Products Development, Warwick Chemical Co. Div., Sun Chemical Corp.

NYLON was introduced in the fall of 1938. Rapid acceptance of hosiery made from it was due to the unique properties which render this yarn almost ideal for hosiery. Among them may be listed tenacity, elasticity, loop resiliency, recovery after stress removal and moisture impermeability.

Unfortunately these very properties which make for excellent, serviceable finished hose, cause serious difficulties in manufacturing.

It is the function of a size to overcome these difficulties, to so improve the physical properties of the yarn that it may be amenable to knitting and other operations.

I shall first summarize briefly the outstanding physical properties of nylon yarn. Then we shall see how these properties affect the operations of hosiery manufacture. Finally I shall describe tests which we have found useful in laboratory evaluation of nylon sizes.

Physical Properties of Nylon

By variations in the degree of cold drawing, nylon fiber can be manufactured with tenacity from 4.5 to 8.0 gm/denier. Thus nylon ranks as one of the strongest fibers known.

As might be expected from its high tenacity, nylon fiber can not be stretched as far as other common yarns before breakage. When we compare nylon, particularly in the wet state, with other fibers used in hosiery manufacture, we find it has only 60 to 80% of the extensibility of other fibers.

Another property almost classing it alone among hosiery yarns is its very high ultimate recovery after stress removal. I say ultimate recovery, because recovery is by no means instantaneous. After its initial, immediate recovery, the yarn continues to contract gradually until it reaches practically 100% recovery.

Nylon yarn is likewise outstanding with regard to its stiffness, being 25% to 200% stiffer than other hosiery yarns.

Considering the high tenacity of nylon, it is surprising that it can be stretched moderately with only slight tension. Unsized nylon requires only one-fourth to one-third as much tension for small amounts of stretch as does silk.

Nylon yarn is further characterized by extremely low moisture absorbency. The fiber is cylindrical and has an extraordinarily smooth surface.

Thus nylon yarn has unusual properties. It has high tenacity, yet requires little tension for low to moderate extensions. Whatever extension does occur in the yarn when subjected to tension, is more or less eliminated after the tension is released.

If we now consider the properties of nylon yarn in relation to fabrication of hosiery, we shall see how these properties affect the several steps of manufacture.

First I mention the one desirable property of nylon which somewhat compensates the hosiery manufacturer for its undesirable qualities. This is the high tenacity of both the dry and wet yarn. It can be subjected to unusually high tensions without breaking in the various manufacturing steps such as sizing, twisting, coning and knitting. In fact, its high tenacity often gives some throwsters and knitters a false sense of security, leading to careless control of tension in process; this in spite of the fact that tension control is of utmost importance with regard to creep or contraction of the knitted fabric. I shall discuss this at great length in a moment.

The relatively low extensibility of nylon yarn, coupled with its high breaking strength could also be a great asset, but to throwsters and knitters the effect of low extensibility is almost completely nullified by high stretchability and complete recovery after stress removal. Low extensibility means that nylon yarn can be stretched only about 18 to 25% before it breaks; but it can be stretched moderately with only slight tensions, and what is more, the yarn will return to its original length after the tension is removed. The resulting difficulties in hosiery manufacture are two-fold. First, in finished hose, non-uniform boot and foot sizes result from non-uniform application of tension on different lots of yarn during the several treatments, with consequent variations in residual shrinkage. Secondly, excessive curling at the seaming machines is due in part to excessive yarn tension during processing, followed by contraction after knitting.

Another physical property of the yarn which affects knitting is the high loop resiliency of nylon fibers. This results, first of all, in poor loop formation; second, it gives distorted stitches; and third, with twisted yarn, it results in a tendency for loops to turn out of the plane of the fabric, yielding a fabric of poor appearance with tendency to snag.

Finally, the property of nylon yarn which gives the sizing chemist most headaches is its general imperviousness. It has low moisture absorbency, a very slick, cylindrical surface, and very poor adherence to most film forming materials which might be used for sizing.

Sizing of Nylon

This was the sad picture we received when first introduced to the problems of nylon hosiery production. Nylon yarn behaves like an elastic band, and is supposed to be handled as such. Beyond certain limits this is of course impossible. The problem then resolved itself into temporarily altering certain of the intrinsic properties of yarn with a size so that it could be converted satisfactorily into hosiery.

When using the polyvinyl alcohol type of size, throwsters generally apply only 3.5 to 4.5% of solids to the yarn. They cannot apply more, because it would adhere poorly and flake off, doing more harm than good. However, there is much evidence to indicate that a higher percentage of sizing solids would eliminate,

* Presented at Hosiery Group Meeting, Augusta National Convention, October 22, 1948.

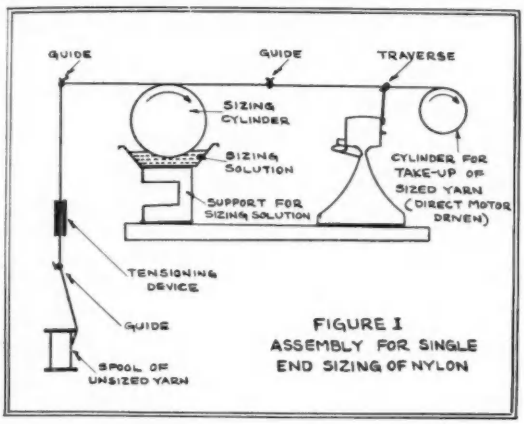

FIGURE I
ASSEMBLY FOR SINGLE END SIZING OF NYLON

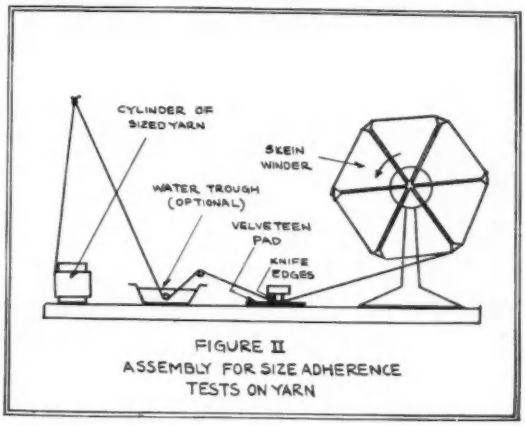

FIGURE II
ASSEMBLY FOR SIZE ADHERENCE TESTS ON YARN

or greatly reduce, many of the difficulties now associated with nylon hosiery production. Because of the characteristics of nylon already described, we felt that 10 to 20% of sizing solids would be more suitable. Yet most of the commonly used sizing materials will not adhere to nylon in amounts exceeding 3 to 4%.

Testing Sizes

Therefore, in laboratory evaluation of a new sizing material, we always test adherence first. This obviously is a prerequisite, for regardless of any other desirable properties, a sizing material will be quite worthless unless it sticks well to the yarn.

We have found the following test methods suitable for estimating adhesive properties of nylon sizes. A rough screening test is first performed by noting adherence of the size to a sheet of nylon. For this we apply a few drops of the sizing solution to a nylon sheet, first scrupulously cleaned with hot water, alcohol and ether. After the size has dried for 48 hours at room temperature, we test adherence by probing the deposited film with a pointed instrument. Very poorly adherent sizes will often be found curled away from the nylon. Slightly adhering sizes will be found stuck to the nylon sheet, but with gentle probing the entire film may be lifted away. Strongly adherent sizes will require firm probing to remove them from the sheet of nylon; what is more, a strongly adherent size will come away from the sheet only in the immediate vicinity of the probe.

If an experimental size shows promise in the sheet-adherence test, we then subject it to further tests, all of which are performed on yarn sized with the material under investigation.

It might be well to describe here the apparatus which we use for single end sizing. It would of course be preferable to use a regular single head knitting-yarn sizer. We didn't have one, so we had to improvise. We apply size by drawing yarn tangentially over the lower roll of our laboratory padder. This roll is stainless steel, and its speed of rotation may be varied over a wide range. The roll is immersed in the sizing solution to a depth of 3 to 4 millimeters. After contacting the sizing roll, the yarn is wound onto a perforated, shellacked cardboard cylinder turned by a high speed laboratory motor. We rigged the parts of an oscillating electric fan to give level winding on the takeup cylinder. The sized yarn is allowed to dry on the cylinder for 48 hours under standard conditions before testing. It may be easier to visualize this apparatus through the schematic drawing in Figure I.

It usually is necessary to run one or two preliminary tests to determine the correct speed of sizing roll in order to get desired size pick-up. We generally prefer to run initial tests on yarn with approximately 12% of size solids. It takes approximately 20 to 30 minutes to size 20 grams of 30 denier yarn when the take-up cylinder runs at 1000 r.p.m. Of this yarn, we use approximately 10 grams for the boil-off to determine the amount of solids deposited, and the balance for evaluating the size.

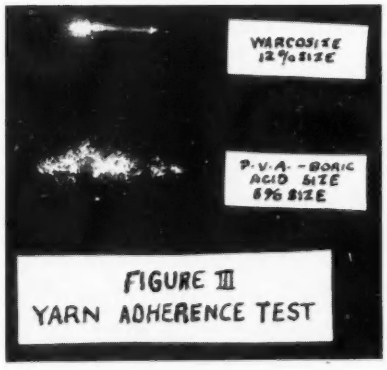

FIGURE III
YARN ADHERENCE TEST

Assuming the boil-off test has shown the desired amount of size deposited, we then proceed as follows:

We first perform a yarn adherence test. This is done by reeling a portion of the sized yarn on a skein winder. As the yarn is drawn from the cylinder to the winder, it passes under a set of knife edges which rests on a pad of dark-colored velveteen. This test is illustrated in Figure II.

It is preferable to have the skein winder motor driven so that it will always turn at the same speed. A dial counter on the winder facilitates running the same number of turns for each test. We find that thirty turns on our skein winder gives satisfactory results.

After the yarn has been drawn through, the knife edges are carefully lifted, and size which has been scraped off upon the velveteen pad is examined. The quantity as well as the quality of the deposit is noted. An ideal size will not, of course, scrape off at all, and although this ideal state is never actually obtained in practice, a good size leaves only a very slight deposit. What is more, the deposit from a good size will be essentially fine powder, free from large flakes and slivers. Flakes and slivers account in large measure for needle clogging in knitting. Tests of several sizes may be run side by side on the pad so that deposits from all may be more readily compared. For the time being we have adopted the commonly used polyvinyl alcohol-boric acid mixture as the standard for evaluating performance characteristics of new sizes. The results obtained in one set of yarn adherence trials are reproduced in Figure III.

It is sometimes advisable to pass the yarn through a trough of water placed in front of the knife edges, so that adhesive properties of the wet size may be observed.

Another test which indicates the ad-

(Concluded on Page P377)

Mid-West Section—

Low Temperature Wool Piece Goods Scouring With A SYNTHETIC ORGANIC DETERGENT*

Laboratory Evaluation

O. M. MORGAN and J. E. WALTER

New Products Division, National Aniline Division,
Allied Chemical and Dye Corporation, Buffalo, N. Y.

Abstract

Wool piece goods containing mineral oil lubricants may be scoured over a wide temperature range with "Nacconol" NR and suitable salts, such as sodium chloride or sodium bicarbonate. Although the quantities of supplies needed are larger at the lower scouring temperatures an over-all economy may be effected when savings in steam, time and labor are considered.

THE removal of emulsified mineral oil lubricants from wool piece goods at the usual scouring temperatures of 100-120° F. may be accomplished easily with a suitable synthetic detergent in conjunction with neutral or slightly alkaline salts such as sodium chloride or sodium bicarbonate. Details of this process were presented in a previous paper *(1)*. The present study examines the performance of these scouring agents at lower temperatures under laboratory scouring conditions.

Cold scouring processes for wool piece goods are not new but are currently demanding more attention as fuel and time savings become more important. Cold scouring processes have been made much more simple and more effective through the use of suitable synthetic detergents which operate efficiently at these lower temperatures. Since the synthetic detergent will rinse much more rapidly than soap and since it may be used in conjunction with neutral or slightly alkaline builders which also exhibit free rinsing characteristics, it can be expected that a very rapid process will result.

The present laboratory scouring experiments were designed to provide guiding data for the mill and were confined to the study of "Nacconol"† NR. These laboratory data have been confirmed, in part, by informal mill reports. Additional mill work is planned to complete this investigation.

Fabric Tested

Wool blanket material weighing 8.5 ounces per square yard and containing 5.7% of an emulsified mineral oil lubricant was selected for this work. This fabric was obtained from a production lot in a woolen mill and for experimental purposes was cut into swatches 3 x 16 inches. The average weight of these swatches was 9.1 grams. Preliminary soxhlet extraction with carbon tetrachloride indicated excellent uniformity in oil content from swatch to swatch.

Laboratory Scouring Procedure

The scouring procedure was essentially the same as that described in a previous paper *(1)*. The scouring solution was prepared at the desired temperature with distilled water and one swatch of fabric agitated gently by hand for 30 seconds. The fabric was then passed between the rolls of an ordinary household wringer, the wringer liquor being returned to the scouring solution. This scouring and squeeze roll procedure was repeated for a total of 10 times and served to simulate the action of either a Dolly washer or a continuous scouring machine.

Rinsing was conducted in two consecutive portions of distilled water at the same temperature as that used for scouring, each rinse being followed by passage of the fabric through the squeeze rolls.

When the scouring and rinsing operations were completed, the fabrics were dried for 3 hours in a forced draft oven at 180° F., following which they were subjected to Soxhlet extraction with carbon tetrachloride to determine their residual oil content.

The major portion of the present work was conducted at low concentrations of synthetic organic detergent in order that the effect of the neutral or alkaline builders might be observed more rapidly without processing large quantities of fabric. Preliminary single scouring tests were conducted where only one piece of fabric was scoured. This was for purposes of selecting the more desirable combinations of scouring supplies. The maximum scouring capacity of these solutions was then studied with respect to the amount of goods that could be processed through a single portion of scouring liquor and still obtain satisfactory residual grease values. These are termed exhaustion tests.

Preliminary Scouring Tests—Effect of Temperature

Earlier laboratory wool piece goods scouring studies *(1)* had indicated that excellent results could be obtained at 130° F. with this synthetic detergent and suitable neutral or mildly alkaline builders. These data were confirmed in full scale mill results in a temperature range of 110-120° F. There were some indications from both laboratory and mill work that lower scouring temperatures could be used successfully and that the selected builders had a very pronounced effect in making this possible. To obtain more information on this subject a series of laboratory scouring tests was conducted at 10° F. intervals over a temperature range of 50-120° F. using 0.5% synthetic detergent solutions with and without 0.5% sodium chloride additions. These data are presented in Table I. Here it will be noted that to obtain a residual grease content of less than 1% a temperature slightly higher than 100° F. is necessary when the synthetic detergent is used by itself. When sodium chloride is used as a builder in the amount indicated, satisfactory scouring may be achieved at 50° F. or probably at a slightly lower temperature.

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* Presented at meeting, Mid-West Section, February 26, 1949.

(1) "Wool Piece Goods Scouring with A Synthetic Organic Detergent". Am. Dye. Reptr., 37, 3, 93-95 (Feb. 9, 1948).

† Trademark "Nacconol" Reg. U .S. Patent Office by Allied Chem. & Dye Corp.

TABLE I

Wool Piece Goods Scouring—Mineral Oil—Effect of Temperature and Sodium Chloride—Preliminary Study

Procedure

Volume of Scouring Solution—1500 cc.
Size of Fabric Sample—3" x 16"—5.7% Mineral Oil.
Scouring Procedure—Stir fabric in solution by hand for 30 sec. Pass through wringer. Repeat to total of 10 immersions. Rinse—2 immersions—1 min. each—distilled water—same temperature as scour.

Exp. No.	Temperature °F.	Residual Oil (%) Synthetic Detergent 0.5%	Synthetic Detergent—0.5% + Sodium Chloride—0.5%
1	Blank—Not Scoured	5.7	5.7
2	50	2.14	0.86
3	60	2.14	0.67
4	70	1.87	0.64
5	80	1.71	0.56
6	90	1.42	0.55
7	100	1.01	0.48
8	110	0.89	0.34
9	120	0.85	0.26

TABLE II

Wool Piece Goods Scouring—Mineral Oil—Single Cold Scour—80° F.

1. Effect of Varying Synthetic Organic Detergent Concentration.
2. Effect of Type and Amount of Builder.

Scouring Conditions—Same as in Table I except temperature is constant at 80°F.

Expt. No.	Synthetic Detergent Conc. (%)—	Residual Oil (%) 0.2	0.3	0.4	0.5	1.0	1.5
1	Synthetic Detergent plus	3.25	2.41	2.07	1.77	1.62	1.93
2	0.1% NaCl	1.58	1.08	1.28	1.31	—	—
3	0.2% NaCl	1.16	0.98	0.97	0.97	—	—
4	0.5% NaCl	0.89	0.58	0.46	0.47	—	—
5	1.0% NaCl	0.78	0.41	0.40	0.40	—	—
6	0.1% $NaHCO_3$	2.41	2.01	1.54	1.41	—	—
7	0.2% $NaHCO_3$	1.65	1.34	1.31	1.33	—	—
8	0.5% $NaHCO_3$	0.98	0.85	0.99	1.21	—	—
9	1.0% $NaHCO_3$	0.84	0.81	1.01	1.06	—	—
10	0.1% Na_2CO_3	2.64	2.71	2.53	2.48	—	—
11	0.2% Na_2CO_3	2.64	2.22	2.82	2.64	—	—
12	0.5% Na_2CO_3	1.91	2.02	2.11	2.13	—	—
13	1.0% Na_2CO_3	1.52	1.46	1.59	1.57	—	—

TABLE III

Wool Piece Goods Scouring—Mineral Oil—Single Hot Scour—120°F.

1. Effect of Varying Synthetic Organic Detergent Concentration.
2. Effect of Type and Amount of Builder.

Scouring Conditions—Same as in Table I except temperature is constant at 120°F.

Expt. No.	Synthetic Detergent Conc. (%)—	Residual Oil (%) 0.1	0.2	0.3	0.4	0.5	1.0	1.5
1	Synthetic Detergent plus	3.45	1.59	1.15	0.98	0.88	1.22	1.34
2	0.1% NaCl	1.56	0.58	0.37	0.45	0.46	—	—
3	0.2% NaCl	0.88	0.42	0.28	0.30	0.38	—	—
4	0.5% NaCl	0.46	0.16	0.19	0.21	0.26	—	—
5	1.0% NaCl	0.48	0.14	0.12	0.18	0.23	—	—
6	0.1% $NaHCO_3$	2.03	1.13	0.77	0.80	0.87	—	—
7	0.2% $NaHCO_3$	1.20	0.67	0.81	0.67	0.71	—	—
8	0.5% $NaHCO_3$	0.46	0.38	0.62	0.73	0.77	—	—
9	1.0% $NaHCO_3$	0.26	0.33	0.37	0.53	0.49	—	—
10	0.1% Na_2CO_3	2.62	2.60	2.53	2.30	2.14	—	—
11	0.2% Na_2CO_3	2.02	2.31	2.24	2.33	2.30	—	—
12	0.5% Na_2CO_3	1.08	1.69	1.77	2.24	2.21	—	—
13	1.0% Na_2CO_3	0.91	1.40	1.53	1.50	1.62	—	—

For purposes of comparing cold and hot scouring processes temperatures of 80° F. and 120° F., respectively, were selected. Rinsing operations were conducted at the same temperature as the scouring operation.

The builders chosen for this work were sodium chloride, sodium bicarbonate and soda ash. Although the results obtained in previous work *(1)* at 130° F. with soda ash and synthetic detergent for the removal of emulsified mineral oil lubricants were rather unsatisfactory, it was felt that soda ash should be included in the present work where a greater variety of conditions was to be encountered.

SINGLE COLD SCOUR—80° F.—Table II provides the results for a single cold scour at 80° F. In this work only one piece of fabric was processed through the scouring liquor. Synthetic detergent concentrations from 0.2-1.5% were used alone or with additions of sodium chloride, sodium bicarbonate and soda ash, respectively, at concentrations from 0.1-1.0%. Best results were obtained with 0.2-0.5% synthetic detergent plus 0.5-1.0% sodium chloride. As the synthetic detergent concentration is increased to 0.4% a gradual improvement is noted. The use of higher synthetic detergent concentrations does not appear to be justified in this laboratory work, but in full scale mill practice where larger quantities of oil are encountered the higher concentrations of synthetic detergent do show a benefit.

With sodium bicarbonate the best scouring results were obtained at 0.5-1.0% and at the lower synthetic detergent concentrations, i.e., between 0.2 and 0.4%.

Although the scouring action of the synthetic detergent was improved to some extent with soda ash additions, it was not possible to obtain a residual oil content in the scoured fabric below 1.0%.

SINGLE HOT SCOUR—120° F.—The results of a single hot scour over a wide range of synthetic detergent and builder concentrations at 120° F. are presented in Table III. Here it will be noted that lower synthetic detergent concentrations may be tolerated than at 80° F. and lower builder concentrations are productive of excellent scouring results. Here again the best scouring results were obtained with the synthetic detergent plus sodium chloride. Good results were also obtained with the synthetic detergent and sodium bicarbonate combinations, but slightly higher concentrations of both scouring agents were needed than when sodium chloride was used as the builder. Where 0.5 and 1% sodium bicarbonate solutions were used, it is interesting to note that the best scouring results were obtained at the lower synthetic detergent concentrations.

As a general rule the scouring results obtained with the synthetic detergent and soda ash were poorer than those obtained with the synthetic detergent by itself, with the exception of the results at the lowest synthetic detergent concentration. In this respect soda ash behaved to some extent like sodium bicarbonate where better scouing results were obtained at the lower synthetic detergent concentrations.

Exhaustion Tests

To determine the comparative periods of useful life that might be expected from the scouring solutions 6 consecutive swatches of fabric were scoured through the same solution and their residual grease contents determined. If the scouring capacity of the bath is high, all six swatches will be scoured satisfactorily. On the other hand, if the scouring solution loses its activity rapidly, the residual oil contents of the scoured fabrics will rise. This procedure simulates what might be expected in a continuous scouring machine.

TABLE IV

Wool Piece Goods Scouring—Mineral Oil—Exhaustion Tests—Cold Scour—80° F.

Scouring Conditions—Same as in Table I except that several consecutive strips of cloth are scoured through the same bath, each strip receiving ten immersions.

Expt. No.	Wool Sample No. — Composition of Scouring Bath	Residual Oil (%) 1	2	3	4	5	6
	0.3% Synthetic Detergent plus						
1	0.2% NaCl	0.90	1.02	1.17	1.31	1.53	1.53
2	0.5% NaCl	0.58	0.70	0.79	0.98	1.15	1.24
3	1.0% NaCl	0.39	0.44	0.52	0.65	0.80	0.86
	0.4% Synthetic Detergent plus						
4	0.2% NaCl	0.88	0.83	0.89	0.99	1.14	1.10
5	0.5% NaCl	0.57	0.52	0.56	0.56	0.71	0.72
6	1.0% NaCl	0.39	0.43	0.43	0.51	0.47	0.50
	0.3% Synthetic Detergent plus						
7	0.5% $NaHCO_3$	0.90	0.94	0.98	1.00	1.07	1.16
8	1.0% $NaHCO_3$	0.64	0.72	0.72	0.70	0.90	0.80
	0.4% Synthetic Detergent plus						
9	0.5% $NaHCO_3$	0.99	0.96	0.87	0.91	0.97	0.96
10	1.0% $NaHCO_3$	0.58	0.50	0.46	0.48	0.42	0.53

TABLE V

Wool Piece Goods Scouring—Mineral Oil—Exhaustion Tests—Hot Scour—120° F.

Scouring Conditions—Same as in Table IV.

Expt. No.	Wool Sample No. — Composition of Scouring Bath	Residual Oil (%) 1	2	3	4	5	6
	0.1% Synthetic Detergent plus						
1	0.2% NaCl	0.78	1.78	2.66	3.00	3.32	3.42
2	0.5% NaCl	0.56	1.14	2.08	2.96	3.33	3.53
3	1.0% NaCl	0.48	0.96	1.57	2.83	3.72	4.15
	0.2% Synthetic Detergent plus						
4	0.1% NaCl	0.58	0.54	0.98	1.07	1.84	2.35
5	0.2% NaCl	0.36	0.38	0.46	0.72	0.96	1.50
6	0.5% NaCl	0.22	0.21	0.24	0.37	0.81	1.10
7	1.0% NaCl	0.19	0.23	0.30	0.32	0.41	0.68
	0.1% Synthetic Detergent plus						
8	0.5% $NaHCO_3$	0.45	0.53	0.62	0.74	1.22	1.42
9	1.0% $NaHCO_3$	0.32	0.42	0.38	0.40	0.53	0.88
	0.2% Synthetic Detergent plus						
10	0.1% $NaHCO_3$	1.13	1.13	1.40	1.42	1.14	1.39
11	0.2% $NaHCO_3$	0.59	0.68	0.62	0.74	0.70	0.94
12	0.5% $NaHCO_3$	0.40	0.40	0.40	0.42	0.44	0.42
13	1.0% $NaHCO_3$	0.33	0.28	0.36	0.39	0.33	0.35
	0.1% Synthetic Detergent plus						
14	1.0% Na_2CO_3	0.87	0.90	0.85	0.80	0.77	0.61

TABLE VI

Cost of Heating 1000 Gallons of Water

Steel Tank—6 x 5′ x 4.725′ deep—Not insulated
Volume of water—1000 gal.
Surface exposure—145.5 sq. ft.
Estimated radiation loss—1.5 BTU/sq. ft./°F/hour
Room Temperature—68°F.
Water Supply Temperature—50°F.

Scouring Temp. (°F.)	Steam Required (lbs.) Heating	Holding 8 hrs.	Total	Total	Steam Cost Reduction (%)
80	258	22	280	$0.168	57.2
120	602	52	654	$0.392	—

EXHAUSTION TEST—COLD SCOUR —80° F.—Reference to Table II indicates that 0.3% or 0.4% synthetic detergent plus 0.2, 0.5 or 1.0% sodium chloride provided good scouring results. These combinations of scouring supplies were selected for exhaustion tests, the results of which are presented in Experiments 1-6, Table IV. The scouring solution containing 0.3% synthetic detergent plus 1.0% sodium chloride gave effective scouring results which were improved to some extent by increasing the synthetic detergent concentration to 0.4%. This provided a lower residual grease content in the scoured fabrics and also made possible the reduction of the sodium chloride concentration to 0.5% should such be desired.

The synthetic detergent-sodium bicarbonate combinations were selected in a similar manner in Table II and exhaustion test studies are presented in Experiments 7-10 in Table IV. The best scouring results were obtained with 0.4% synthetic detergent plus 1.0% sodium bicarbonate.

EXHAUSTION TEST—HOT SCOUR —120° F.—From the single hot scour data, Table III, concentration combinations of synthetic detergent and builder were selected for exhaustion tests, the results of which are presented in Table V. Experiments 1-3 in Table V show that 0.1% synthetic detergent in conjunction with any studied quantity of sodium chloride is not sufficient to provide a suitable scouring result on an exhaustion test basis. However, when the synthetic detergent concentration is increased to 0.2% in Experiments 4-7 a satisfactory scouring result is obtained in the presence of 1.0% sodium chloride. By a further increase in the synthetic detergent concentration, lower concentrations of sodium chloride might be tolerated. Evidence of this is contained in Table I, Experiment 9.

The action of sodium bicarbonate in extending the useful period of life of the scouring bath is presented in Experiments 8-13, Table V. Here again it is demonstrated that it is a matter of balancing the synthetic detergent concentration with the concentration of the builder to obtain the best results.

Experiment 14, Table V, presents an exhaustion test for a scouring solution containing 0.1% synthetic detergent plus 1.0% soda ash. A reasonably good scouring action was obtained which was sustained and even improved slightly as the exhaustion test progressed. Full scale mill trials are needed to confirm these results.

Comparison of Cold and Hot Scouring Methods

The laboratory experimental data obtained in the present study indicate that emulsified mineral oil lubricants may be removed satisfactorily from wool piece goods over a broad temperature range when using proper combinations of synthetic detergents and suitable neutral or mildly alkaline builders. In the cold scouring method higher concentrations of scouring supplies are needed than when the hot scouring method is used, but this increase in supplies is not prohibitive.

Through the use of the cold scouring method steam consumption is greatly reduced. An example of what may be expected is presented in Table VI where cost comparisons are given for heating 1000 gallons of water to either 80° F. or 120° F. and holding it at these respective temperatures for an 8-hour period. For purposes of this comparison a steel tank was considered, carrying no insulation. The average water supply temperature, considered on a yearly average basis, was taken as 50° F. while the room temperature in the mill, again taken on a yearly average basis, was considered as 68° F. The number of pounds of steam required to heat the water from 50 to 120° F. for the hot scour and from 50 to 80° F. for the cold scour was then calculated as well as the steam required to hold this temperature for 8 hours in the presence of a room temperature of 68° F. The cost of steam was taken as $0.60 per thousand pounds which is considered to be an average figure at present fuel costs.

It will be noted that by using the cold scour steam costs were reduced 57%.

This reduced steam demand should provide many mills with increased flexibility without additional capital expenditures for new boiler equipment.

In mills where the water is brought up to temperature in the washers less time will be lost when waiting for the water to be heated. This would also be reflected in decreased labor costs.

Although some informal mill data are now at hand concerning the utility of this cold scouring process, further full scale experiments are planned to provide additional data on this subject.

The statements made herein with respect to "Nacconol" are expressions of the author's opinion, based on careful tests and investigations, and are not warranties.

Sizing Materials—

(Concluded from Page P373)

hesive properties of a size, and measures its cohesive power as well, is the filament binding test. To perform this test, the sized yarn is placed in a twist counter and any twist present is removed. While the yarn is still in the counter, a needle is inserted between the filaments and gently forced along the yarn. As the needle spreads the filaments, it should be noted whether any appreciable dusting of size occurs, and whether any flakes or slivers are scraped off. The operator should likewise note how much force is required to separate the filaments. With a little experience he can readily distinguish between a weakly coherent and a strongly coherent bundle of filaments.

As pointed out previously, a size will be worthless unless it adheres to the yarn. By the same token, mere adherence is of no value unless the size can so alter certain of the yarn's physical properties as to make it suitable for knitting and other handling operations. This is the real function of the size.

Let us review briefly what we wish to accomplish by means of the size. We would reduce stretching of the yarn; we would reduce recovery after removal of tension; we would form the loops easier and hold them in the plane of the fabric; we would make a firm, non-curling fabric on which seamers and inspectors can work efficiently, with a minimum of picks; and we would make a fabric which will not wilt and will maintain its shape until set by the preboarding operation.

To determine the stretch and recovery properties of sized yarn, it is advisable to use a Scott yarn tester such as Model IP-2, or some similar instrument. A bundle of yarns, containing a sufficient number of strands to give 300 denier, is placed in the machine; a given tension is applied to the yarn, and the amount of stretch is noted. After say one minute, tension is released. Elongation is noted immediately, then again after ten minutes. The results thus obtained may be compared with results from unsized yarn, as well as from yarn bearing a standard size. An efficiently sized yarn will be characterized by lower elongation under tension, and smaller recovery after tension is released.

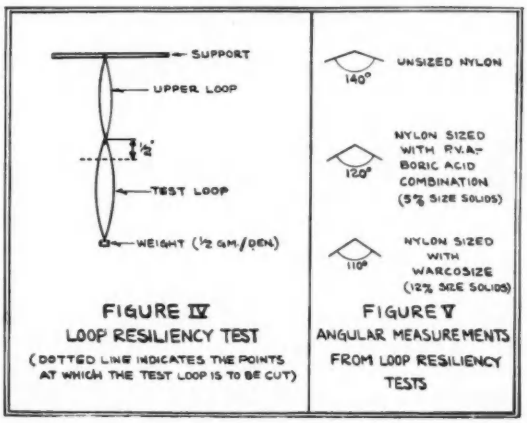

FIGURE IV
LOOP RESILIENCY TEST
(DOTTED LINE INDICATES THE POINTS AT WHICH THE TEST LOOP IS TO BE CUT)

FIGURE V
ANGULAR MEASUREMENTS FROM LOOP RESILIENCY TESTS

We have also run this test roughly, by hanging a weight, approximately 3 gram/denier, to the end of a bundle of yarns of given length; noting the stretch and then the recovery after removal of the weight. The data thus obtained are not of course as accurate as from an instrument designed for the purpose.

The loop forming properties of the yarn may be determined by the following simple test *(1)*. A loop of yarn is hung from a support. Through this loop is placed a length of the yarn being tested. The ends are knotted to form another loop. From the knot of the lower test loop a small weight is hung, approximately ½ gram/ denier. After hanging thus for 30 seconds, the strands of the lower loop are cut one-half inch from the top. The resultant one inch length of yarn left hanging in the upper loop will form an angle, which will be smaller or larger depending on the efficiency of the size. Thirty seconds after cutting, the angle is measured by means of a small protractor. Unsized nylon shows an angle of approximately 140 degrees. Sized yarn which will knit with good loop formation shows an angle of 120 degrees or less; the smaller the angle the better. For accurate results this test should be conducted under standard conditions of temperature and humidity, and at least 10 determinations should be run on each sample for a reliable average. Figure IV illustrates this loop resiliency test, and Fixure V shows the results obtained in one series of tests.

Limp yarn tends to cling to the needles when the formed loops are slipped off through the new loops. If loops do not slip from the needles smoothly, poor stitch formation or "press-offs" result. A limp fabric slows down the seaming operation and tends to pick badly. Therefore it is generally advisable to get as firm a knitted fabric as practical, all other factors being equal. The firmness quality of sized yarn, as well as of knitted fabric to be obtained from it, may be roughly estimated by preparing a skein from the sized yarn and comparing its hand with that of a similar skein unsized, or treated with a standard size. As knitted fabric may be stored several weeks before seaming, or seamed hose may be stored before boarding, it is also important to determine how ageing will effect firmness of the fabric. It should not creep or wilt on ageing. For this test skeins of yarn are treated with the experimental size, the standard size, and with no size. Without undue handling these skeins are stored for two weeks under conditions of temperature and humidity which might be expected in hosiery mills. These skeins are then tested for hand in comparison with similar, freshly prepared skeins, as to whether the aged skeins have as much body as the fresh.

In final analysis, the only sure test for a new yarn size is actual hosiery manufacture. There are certain factors which can be evaluated in no other way. But in developing WARCOSIZE our laboratory has been able to save time and effort, and to avoid blind alleys, by using the test methods described.

Acknowledgment

Grateful thanks are offered to Mark Holmes, Henry Dixon and Aaron Burdison for their invaluable assistance.

Reference

(1) M. N. Holmes, "Underwear and Hosiery Review", pp. 97-99, December, 1941.

Mid-West and Philadelphia Sections—

Studies on Wool Dyeing

EFFECT OF METALS ON TIPPY DYEING*

HENRY E. MILLSON

American Cyanamid Company, Calco Chemical Division, Bound Brook, New Jersey

Introduction

SHRINKAGE control of wool fabrics of all kinds has created an unprecedented demand for materials which have been colored with dyes capable of withstanding repeated launderings without losing color body or suffering distinct shade changes. One of the methods of obtaining dyed wool material having satisfactory fastness properties, brightness of hue and solidity of shade is the use of chrome colors which are ordinarily applied by either the top or the meta-chrome dyeing procedures. Consequently, the problem of obtaining solid, reproducible shades by either of the chrome dyeing methods has become increasingly important. It is believed that much of the difficulty in obtaining solid shades with chrome dyes is due to tippy dyeing and that a remedy may be found through the further study of the causes of tippy dyeing.

Analyses of mill problems concerning chrome dyeing methods have shown that there are many conditions which can cause wide variations in shade. Among the conditions most difficult to control are those caused by the presence in the dye bath of the soluble salts of aluminum, copper, and iron, the latter being the most frequent offender. Such metals may enter the dye bath in several ways. They may be soluble salts carried in by natural or treated waters and by the rusting of water pipes; they may be dissolved from machine parts, linings and pipes during the dyeing process. Several excellent articles on the sensitivity of chrome dyes to iron have been published recently *(1, 2, 3, 4, 5)*. However, the effect of the presence of aluminum and copper on the shade and fastness properties of chrome dyes has received very little attention. In general, chromable dyes are polygenetic and react readily with various metals to give different shades. Consequently, the presence of traces of alumnium, copper or iron in a meta, top or bottom chrome dye bath might permanently alter the shade of the dyeing. Iron salts generally cause browner and duller shades, whereas copper salts usually cause redder and duller shades. Salts of alumnium might cause definite shade changes ranging through bluer, redder or violet tones.

The present study has been concerned chiefly with the effect of metals on the deposition and distribution of dye within the individual fibers. The distribution of trace metals in the fiber has been determined by cross-sections and other microscopical studies. The rate and extent of metallization by various metals have been studied by means of the spectrophotometer. It has been observed that the presence of metals in the dye bath is an important factor in two of the major dyeing difficulties. It may not only cause the wool to dye tippy, but it may also frequently be the reason for undesirable color changes during the dyeing procedure. It has also been observed that the extent of damages in a staple or part of the fleece varies considerably, depending upon its original location in the fleece. In this study, wherever possible, only staples from the back and shoulder areas have been used.

Metallization of Chromable Dyes

Dyes which can react with chromium when dyed by one of the various chrome dyeing procedures may also react with many other metals, such as aluminum, cobalt, copper, iron and nickel, to name but a few. These metal-dye complexes may exhibit wide differences in shade and fastness properties depending upon the metal complex which is formed. The first part of this study will describe a number of experiments on the metallization of dyes. Several of these experiments are described in detail because they are useful in explaining some of the results presented later in the tippy dyeing studies.

COLOR OF METAL-DYE COMPLEXES.—Figures 1-4 are spectrophotometric curves for 0.5% dyeings of Calcochrome Fast Red ECB, C.I. 652; Calcochrome Alizarine Red SC, C.I. 1034; Calcochrome Cyanine RC, C.I. 722; and Calcochrome Violet B, C.I. 169, respectively. The dye bath was prepared with 10% calcined Glauber's salt, and 5% acetic acid 28%, in each instance, and after the skeins were entered they were brought to the boil slowly and dyed at the boil for 1 hour. The liquor-wool ratio was 60:1. One such dyeing of each was rinsed and dried, whereas others were boiled separately for 30 minutes in a fresh bath, with an addition of 2% acetic acid 28%, and 0.75% of one of the following salts:

Aluminum sulfate $Al_2(SO_4)_3 \cdot 18\,H_2O$
Potassium bichromate, $K_2Cr_2O_7$
Copper sulfate, $CuSO_4 \cdot 5H_2O$
Ferrous sulfate, $FeSO_4 \cdot 7H_2O$

A comparison of the curves shows that each metal produced an entirely different shade with the same dye. Consequently,

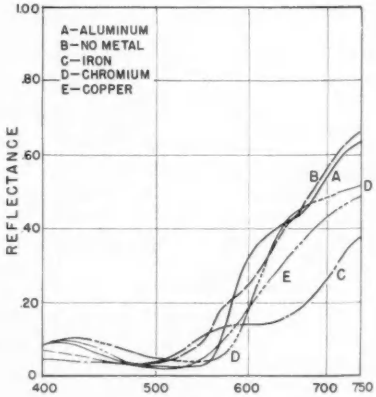

FIGURE 1

Effect of Metals on Calcochrome Fast Red ECB 0.5% dyeings of Calcochrome Fast Red ECB on wool were made by the acid dye procedure and after-treated with 0.75% metals as indicated.

* Presented at meetings of the Mid-West Section, September 25, 1948 and the Philadelphia Section, March 11, 1949. At the presentation of this paper in Chicago, 160 Kodachrome slides were used. Four projectors and four screens were used so that slides showing a staple of fleece, a longitudinal section of the tip and body parts of a fiber, a cross section of the tips, and a cross section of the body parts of the same fibers could be projected simultaneously for comparison.

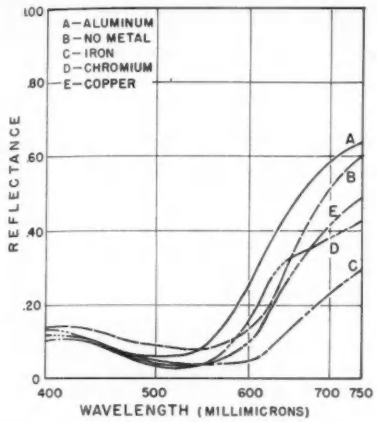

FIGURE 2

Effect of Metals on Calcochrome Alizarine Red SC

0.5% dyeings of Calcochrome Alizarine Red SC on wool were made by the acid dye procedure and aftertreated with 0.75% metals as indicated.

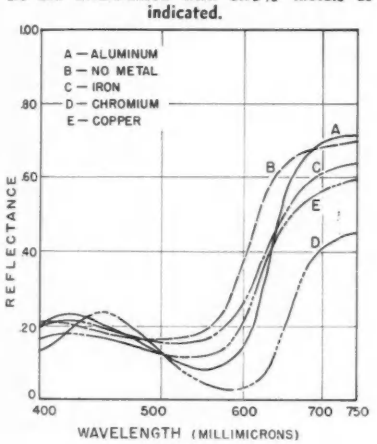

FIGURE 3

Effect of Metals on Calcochrome Cyanine RC

0.5% dyeings of Calcochrome Cyanine RC on wool were made by the acid dye procedure and aftertreated with 0.75% metals as indicated.

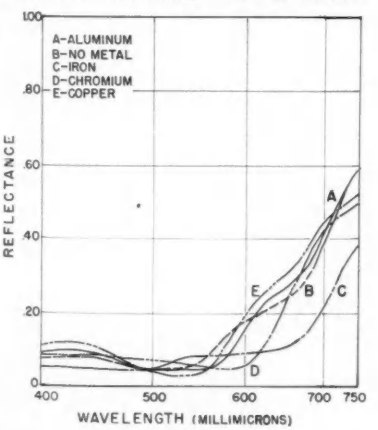

FIGURE 4

Effect of Metals on Calcochrome Violet B.

0.5% dyeings of Calcochrome Violet B on wool were made by the acid dye procedure and aftertreated with 0.75% metals as indicated.

traces of these elements may affect the final shade of a chrome dye in proportion to the amount of metal which is present. These trace metal salts are especially detrimental in a top or bottom-chrome dye bath because they form metal-dye complexes rapidly and at comparatively low temperatures, before the dye can react with the chromium in the wool in bottom-chrome dyeing or prior to the addition of bichromate in top-chrome dyeing. Except in the case of aluminum, the undesired metal in the molecule is not replaced during the top-chroming process after the metal complex has once been formed, regardless of the amount of bichromate added, as can be seen in the following experiment.

The purpose of this experiment was to determine the effect of afterchroming on dyeings which had previously been metallized by topping with aluminum sulfate, cobalt acetate, copper sulfate, ferrous sulfate or nickel sulfate. These dyeings were made as above except that 1% of Calcochrome Violet B was used. One skein was removed and dried while another was placed in a fresh bath containing 1% of bichromate and 3% of acetic acid 28% and boiled for an additional 40 minutes. The amounts of acid, metal salt, and the dyeing time were deliberately increased over the quantities used in the experiment described in the preceding paragraph to see if chromium would replace the other metals. A comparison of the dyeings before and after the treatment with bichromate showed that no change occurred with the dyeings which had been topped with copper, cobalt or nickel in the dye bath. A dyeing in which iron had been used was slightly redder in shade. The dyeing topped with aluminum was a rich fuchsia shade before top-chroming but it became reddish violet when it was afterchromed, showing that the chromium appeared to replace, in part, the aluminum in the aluminum-dye complex.

Although various dyes exhibited their own peculiarities, in general, the cobalt, copper, iron, and nickel dyeings changed very little in after-chroming. The effect of aluminum, however, was overcome to a considerable extent by the bichromate treatment. Further experiments were run to determine the effect on the shade when equal amounts of bichromate and another metal salt were added simultaneously to the top chroming bath. A liquor ratio of 60-1 was used for all dyeings unless stated otherwise. Calcochrome Brown PG, Pr. 4, Calcochrome Brown RV Conc., Pr. 2, and Calcochrome Violet B, were used for these studies. One per cent of dye was used with an addition of 10% calcined Glauber's salt and 6% acetic acid 28%. Each dyeing was boiled for 45 minutes, then it was topped in the dyebath with 1% cobalt acetate, nickel sulfate, copper sulfate or aluminum sulfate. A second series was also carried out in the same manner except that 1% potassium bichromate was added with the metal salt in all cases.

Examination of the Brown PG dyeings showed that the cobalt, copper and iron dyeings gave approximately the same shade whether or not bichromate was added, indicating that with this dye these metals react rapidly to the exclusion of the chrome. The presence of nickel and aluminum caused a slight effect on the shade but the chromed shade predominated. With Calcochrome Violet B, the bichromate had a much greater effect on the shade in all cases. The shades obtained, however, were nearer to those produced by the metals other than chrome. The aluminum dyeings were an exception to this general statement, since the dyeing topped with aluminum and bichromate yielded a slightly redder chromed shade. The Brown RV dyeings showed some effect of the chrome in the cobalt and nickel dyeings only, but in these instances the shades of these metal complexes predominated.

EASE OF METAL-DYE COMPLEX FORMATION.—Since Calcochrome Brown RV Conc. is known to form metal complexes with a number of metal ions it was used in a series of experiments to illustrate the metallization of a dye in solution. This study was carried out by means of the spectrophotometer. An 0.05% aqueous solution of the commercial dye was prepared with distilled water. A spectrophotometric transmission curve of this solution was obtained and identified as curve "A." A series of curves was then procured after the solution of various metal salts had been added to the original. For example, Curve "B" was obtained by raising the temperature of the dye solution to 38° C, then adding 5 ml. of a 10% solution of a metal salt, and then waiting 15 minutes before the curve was taken. Figures 5 to 7 represent the curves obtained with Calcochrome Brown RV, using this procedure with aluminum sulfate, copper sulfate and ferrous sulfate, respectively. These tests show that metallization of the dye molecules takes place rapidly at low temperatures, that each metal produces a different shade and that copper sulfate causes the dye to precipitate.

An attempt also was made to determine the effect of the presence of bichromate on this type of reaction. Solutions were prepared as before but 5 ml. of a 10% solution of sodium bichromate ($Na_2Cr_2O_7 \cdot 2H_2O$) was added simultaneously with the metal salt addition. These solutions were again allowed to stand 15 minutes before the transmission curves were taken. The dye solutions containing aluminum,

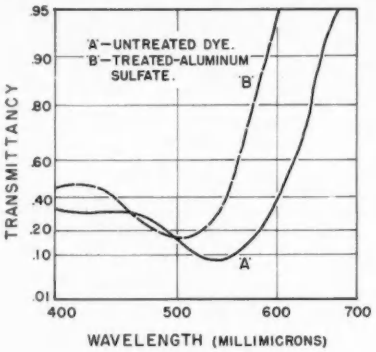

FIGURE 5
Effect of Aluminum on Calcochrome Brown RV Conc. in Solution.
Curve A represents a 0.05% solution of Calcochrome Brown RV Conc. prepared in distilled water. Curve B is the same solution after 0.25% of aluminum sulfate had been added and the temperature raised to 38°C.

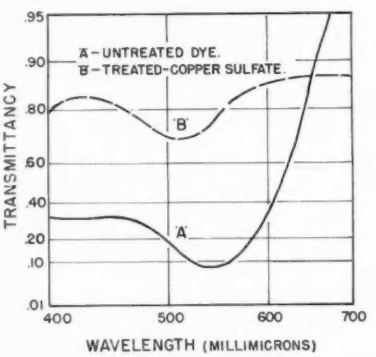

FIGURE 6
Effect of Copper on Calcochrome Brown RV Conc. in Solution.
Curve A represents a 0.05% solution of Calcochrome Brown RV Conc. prepared in distilled water. Curve B is the same solution after 0.25% of copper sulfate had been added and the temperature raised to 38°C.

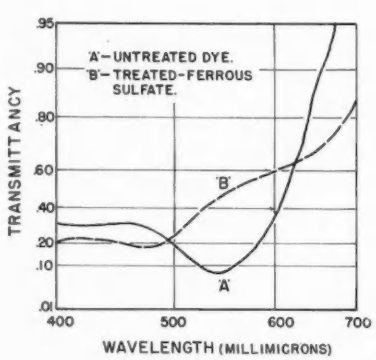

FIGURE 7
Effect of Iron on Calcochrome Brown RV Conc. in Solution.
Curve A represents a 0.05% solution of Calcochrome Brown RV Conc. prepared in distilled water. Curve B is the same solution after 0.25% of ferrous sulfate has been added and the temperature raised to 38°C.

iron and copper became turbid, indicating precipitation, and so curves could not be obtained. The solutions containing nickel sulfate remained clear. However, when the transmission curve of the solution containing nickel sulfate and sodium bichromate was corrected for the equivalent bichromate solution, a calculated curve was obtained which was identical to that found for the dye solution containing nickel sulfate only. Figure 8 shows these curves which, because they superimpose, demonstrate that the presence of the chrome had no effect under the conditions of this experiment. Similarly, figure 9

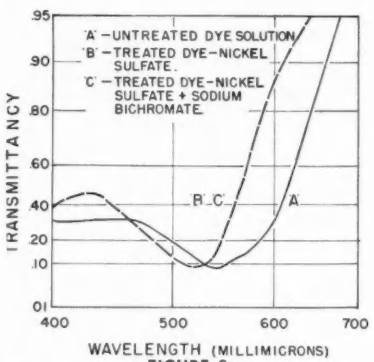

FIGURE 8
Effect of Simultaneous Additions of Nickel and Chromium to Calcochrome Brown RV Conc. Solution.
Curve A represents a 0.05% solution of Calcochrome Brown RV Conc. in distilled water. Curve B shows the same solution after 0.25% of nickel sulfate had been added and the temperature raised to 38°C.
Curve C shows solution A after 0.25% of nickel sulfate and 0.25% of sodium bichromate had been added and the temperature raised to 38°C.

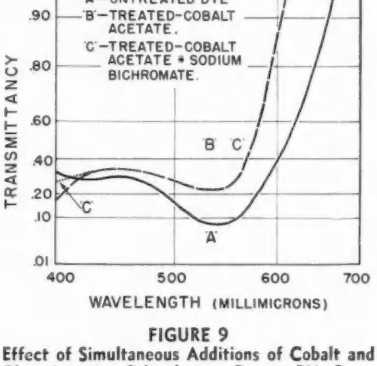

FIGURE 9
Effect of Simultaneous Additions of Cobalt and Chromium to Calcochrome Brown RV Conc. Solution.
Curve A represents a 0.05% solution of Calcochrome Brown RV Conc. in distilled water. Curve B shows the same solution after 0.25% of cobalt acetate had been added and the temperature raised to 38°C.
Curve C shows solution A after 0.25% of cobalt acetate and 0.25% of sodium bichromate had been added and the temperature raised to 38°C.

shows that the cobalt and cobalt-bichromate curves are identical except for a very slight deviation in the blue part of the curve.

SOURCES OF METAL CONTAMINATION.—Interfering metals can be introduced into a dye bath in many ways. Dyes may contain these metals as impurities, despite the fact that manufacturers of dyes have set up rigid controls for the purification of intermediates and for careful selection of construction materials in order to eliminate or reduce to a minimum the amount of aluminum, copper, iron or other metals in their products.

Dyeing machines may act as sources of contamination, although modern dyeing machines are generally constructed of stainless steel and will not cause serious metal effects. However, some machines still in use do contain one or more metals, such as aluminum, copper, bronze, brass or iron. Other machines with Monel metal or stainless steel linings are fitted with iron spiders, bronze and brass segments, copper nails and springs and iron piping. These metals are potential sources of trouble if they are dissolved in the dye bath.

Dyeing accessories such as dye and acid pails, dippers and brass spigots may be attacked by acid or ammonia. Ammonia, sometimes used for scouring or dyeing wool, may be run through a copper or brass spigot into a copper bucket. The ammonia in this case is generally bluish in shade, indicating the presence of dissolved copper.

Probably one of the greatest unsuspected contributors to contamination is the water used for dyeing. Invariably, it contains traces or larger amounts of metallic compounds; this is especially true of iron, which is almost always present in steam and water pipes, either as iron rust or as soluble iron compounds. Trouble is particularly apt to occur when the valves are first opened after having been closed overnight or over a week-end. It is a wise precaution to move the pipe out of the dye kettle and run the water down the drain for several minutes before using it to fill the machine. In this way, most of the accumulated dirt, iron and rust are flushed away. Ground and well waters frequently contain iron and should be analyzed periodically to check the quantity present. Other elements such as aluminum, calcium and magnesium are to be found in water and tend to contaminate the dye bath. Alum is often used in the purification of municipal waters and any excess which remains in the water is a source of aluminum which causes greater shade changes than either calcium or magnesium compounds.

Some chemicals used in the dye bath may act as a source of contamination.

Glauber's salt, for example, varies considerably in its iron content. The better grades now are so processed that they contain none or at most moderate amounts of impurities. However, prior to World War II, some of the cheaper grades of crystalline Glauber's salt were found to contain comparatively large amounts of iron. Other chemicals used in the dyeing process should be carefully evaluated for their impurities, particularly for the presence of iron and copper.

When all of these contaminating sources are taken into consideration, it is easy to understand that wide variations in shade from batch to batch are likely to occur, particularly if the dyes are sensitive to these metals.

SENSITIVITY OF DYES TO METALS. —The concentration of metals such as aluminum, copper and iron, which are generally present in water in small amounts, is expressed in parts per million, or p.p.m. Experiments have shown that many widely used chrome dyes are extremely sensitive to iron and copper, and that their shade may be completely changed by the presence of 1 or 2 p.p.m. of these metals in the dye bath. As stated earlier, the color changes caused by iron compounds are generally detrimental because the iron complexes of most dyes are brown. This has the same dulling effect upon a shade as the addition of small amounts of brown or other dulling dyes to bright contrasting shades. To illustrate the effect of iron Figure 10 shows a family of curves produced by dyeing 0.5% of Calcochrome Fast Red ECB with 8% calcined Glauber's salt, 3% acetic acid 28%, in distilled water, to which is added 0.0, 0.1, 0.25, 1, 3, and 5 p.p.m. of iron respectively.

Other metals also cause shade changes. Figure 11 shows a family of spectrophotometric curves obtained from dyeings made as above with 0.5% Calcochrome Blue Black Conc., but with an addition of 1 p.p.m., 3 p.p.m., and 5 p.p.m., of copper, respectively. Figure 12 shows a series of curves produced by dyeing 0.5% Calcochrome Alizarine Red SC plus 0.0, 0.1, 0.25, 1, 3, and 5 p.p.m. of aluminum respectively. These curves show that the presence of 1 part of aluminum in 10,000,000 parts of water can change the shade sufficiently to produce a measurable variation in the spectrophotometric curve of the dyeing. Figure 13 illustrates the difference in the shapes of the curves obtained from 0.5% dyeings of Calcochrome Fast Green SGV with additions of 0.2, 1, and 2 p.p.m. of cobalt as compared to the "no-addition" dyeing.

To assist in understanding why one or two parts per million of metal can cause alteration of shades, information is given in Table 14 for two well-known chrome dyes.

From this data then, it can be seen that the proportion of pure dye converted to the metal complex by 1 part of metal ranges from 6.5 to 16.5 parts. A dye bath containing 1% of pure dye at a bath ratio of 1-60 contains approximately 166 p.p.m. of dye. In general, commercial dyes are rarely pure because of the dilution necessary in standardization. For this reason, the actual concentration of pure dye may be much less than 166 p.p.m., while the undesirable color change caused by 1 part of metal will be inversely proportional to the amount of pure dye present in the dye bath. In top chrome dyeing, the combination of metal with dye is generally one atom of chromium with two molecules of pure dye. If it is assumed that one atom of iron combines with one molecule of dye, then when Calcochrome Fast Red ECB with a purity of 50% is applied by the top chrome procedure, 1 p.p.m. of iron in a liquor ratio of 60-1 will convert approximately 10% of the pure dye to the iron complex. Under the same conditions, 1 p.p.m. of aluminum would metallize approximately 20% of this dye. Table 34 shows that Calcochrome Fast Red ECB

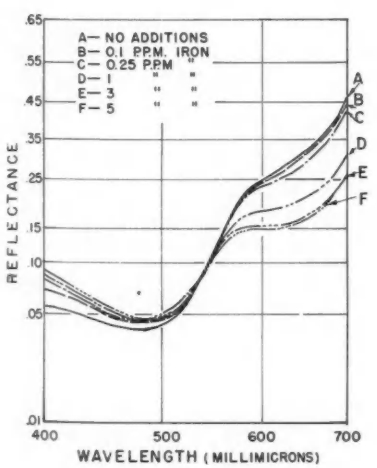

FIGURE 10
Effect of Concentration of Iron on Calcochrome Fast Red ECB.
Dyeings on wool were made by the acid dyeing method of 0.5% Calcochrome Fast Red ECB with the addition to the dye bath of the various amounts of iron indicated on the curve.

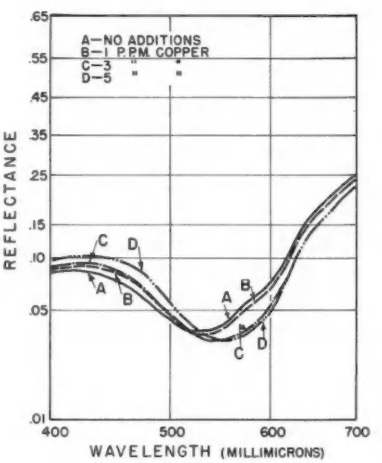

FIGURE 11
Effect of Concentration of Copper on Calcochrome Blue Black Conc.
Dyeings on wool were made by the acid dyeing method of 0.5% Calcochrome Blue Black Conc. with the addition to the dye bath of the various amounts of copper indicated on the curve.

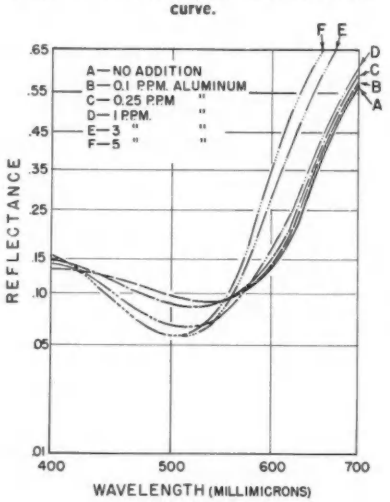

FIGURE 12
Effect of Concentration of Aluminum on Calcochrome Alizarine Red SC
Dyeings on wool were made by the acid dyeing method of 0.5% Calcochrome Alizarine Red SC with the addition to the dye bath of the various amounts of aluminum indicated on the curve.

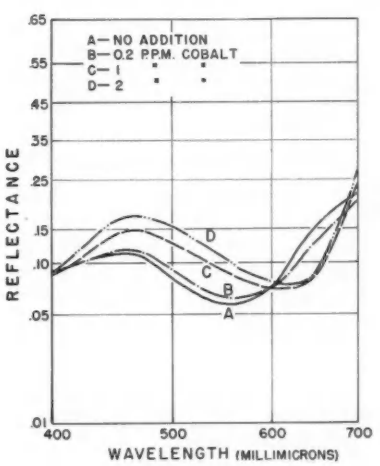

FIGURE 13
Effect of Concentration of Cobalt on Calcochrome Fast Green SGV.
Dyeing on wool were made by the acid dyeing method of 0.5% Calcochrome Fast Green SGV with the addition to the dye bath of the various amounts of cobalt indicated on the curve.

TABLE 14

1 part by weight of metal	*Metallizes the following parts by weight of pure dye* Calcochrome Fast Red ECB	Calcochrome Blue Black Conc.
Aluminum	16.5*	15.4
Chromium	8.6	8.0
Copper	7.0	6.5
Iron	8.0	7.4

* The data in the above table have been calculated by assuming that one atom of the metal combines with 1 molecule of the dye. It is known, however, that under certain dyeing conditions 1 atom of chromium combines with two molecules of dye. Consequently, the values for chromium in columns 2 and 3 may well be 17.2 and 16 respectively.

gives a bright bluish red with chromium and a medium yellowish brown with iron. Hence, if 10% of the pure dye present is metallized with iron, the dulling effect will be approximately equal to the addition of 10% of brown on the weight of pure Red ECB used. Pastel or light shades are affected to a greater extent than are the heavy shades, since the ratio of metal to dyestuff is proportionally higher. For example, if there is sufficient metallic impurity present in a top chrome dye bath to metallize 10% of the dye in a 1% dyeing, all of the dye might be metallized in a 0.1% dyeing of the same dye.

INFLUENCE OF LIQUOR RATIO.—The liquor ratio obviously plays a very important part in metal effects. Water containing 1 p.p.m. of iron or other metal will give different shades with the same dye, showing a greater effect with longer liquor ratios. This occurs because a dye bath with a liquor ratio of 1-10 will contain only one-tenth as much iron per unit weight of dye as one having a liquor ratio of 1-100, though in each case the iron concentration of the water is the same. Changes in shade caused by different liquor ratios are shown in Figures 15, 16, and 17. These experiments demonstrate that when dye house waters contain dissolved metals, the liquor ratio should be reduced to a minimum in applying dyes which are sensitive to these metals.

Preferential Metallization of Dye in Tips and Other Damaged Areas

TOP CHROME PROCEDURE.—Studies on the preferential chromation of dye in wool fibers have been reported previously *(6)*. In these early experiments, skeins of yarn were dyed, and cross sections of the dyed yarn showed that some fibers appeared to be completely chromed while others remained unchromed when the quantity of chrome used was insufficient for complete chromation of all the absorbed dye. During the present investigation, in which a few p.p.m. of aluminum, copper, iron, or other metals were used, skeins of yarn were also used at the beginning of the investigation. Again it was observed that certain fibers in a cross section would present a shade entirely different from the shade of the surrounding fibers. It was also observed that the shade exhibited by these occasional sections was approximately that produced in 1% dyeings which were topped with 0.75% of the corresponding metal compound. When staples removed from Australian, Texas, or South American fleece were used in place of yarn, it was possible to study the entire length of the individual fibers of which the staple was composed. The reason for the distinct color changes observed previously in certain cross sections then became apparent. It was found that the tips and body portions of the same fleece or fiber exhibited different shades when they were dyed in the presence of a few p.p.m. of different metals. In general, cross sections of the tips and body parts of the same fibers removed prior to top chroming showed that the tips were the shade of the metal-dye complex of the particular metal used, whereas the major part of the staple was the shade of the unchromed dye. After the dyeing had been top-chromed, many of the tips were found to have changed very little during the chroming operation, whereas the body portions changed to the chrome shade.

In the discussion which follows, one or more of three types of experiments may be described. The first type is the dyeing to obtain a "self-shade" in which no metal is used. The second type employs a few parts per million of metals other than chrome, such as aluminum, copper, iron, etc. For example, when the dye and iron are used, the treatment will be designated as "ironed." If copper is used with the dyestuff, the treatment will be described as "coppered." The third type takes some of the ironed or coppered skeins and follows with a standard "top chroming" procedure. The purpose of this was to determine whether or not a metal-dye com-

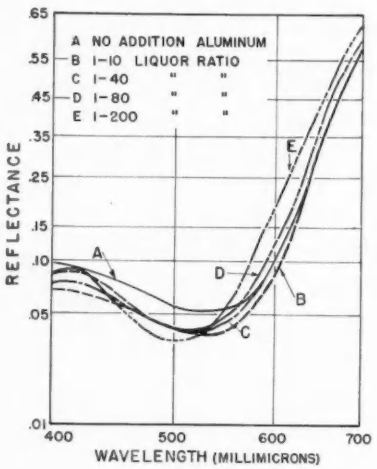

FIGURE 15
Effect of Dye Bath Length on Action of Aluminum on Calcochrome Alizarine Red SC. Dyeings on Wool were prepared by the acid dyeing method using a dye bath containing 1 p.p.m. of aluminum. Different dye bath ratios were used as indicated on the curves.

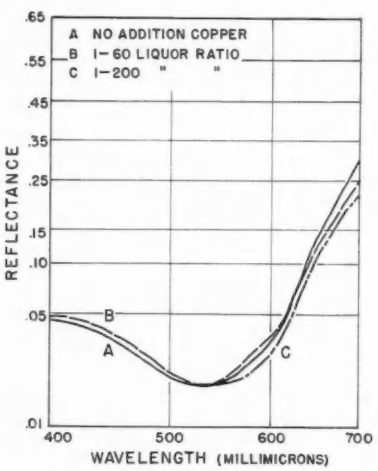

FIGURE 16
Effect of Dye Bath Length on Action of Copper on Calcochrome Blue Black Conc. Dyeings on Wool were prepared by the acid dyeing method using a dye bath containing 1 p.p.m. of copper. Different dye bath ratios were used as indicated on the curves.

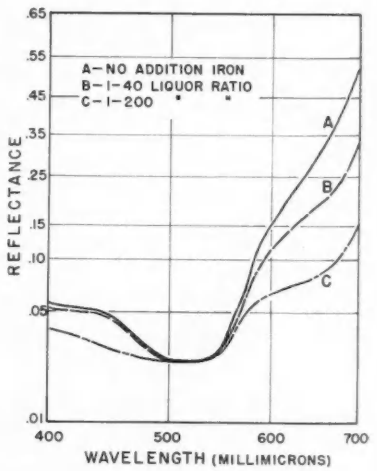

FIGURE 17
Effect of Dye Bath Length on Action of Iron on Calcochrome Violet B. Dyeings on Wool were prepared by the acid dyeing method using a dye bath containing 1 p.p.m. of iron. Different dye bath ratios were used as indicated on the curves.

plex, which might be formed by any trace elements present, was subsequently broken down and a chrome-dye complex formed. Unless this latter occurs, the final shade obtained in a practical dyeing will not be that shade which was anticipated.

Chrome dyes which yield contrasting shades between the acid and metallized dyeings were used because of the ease with which slight differences caused by preferential metallization could be detected. It should be emphasized that, with the exception of Calcochrome Brown RH (C.I. 98) which exhibits a moderate tendency to dye tippy, all of the dyes selected exhibited little tendency to dye tippy when they were applied by the top chrome procedure (7). Therefore, if tippy effects were obtained with these dyes, this tippiness could be attributed to the presence of the minute quantities of the metals which were purposely added to the dye bath. From the results obtained, it is clear that under adverse conditions tippy effects may be produced with dyes which otherwise would not exhibit tippiness.

The fleece used for all tests was a sheared, unscoured, super Australian Merino 64's quality. It was cleaned and prepared for dyeing by extracting several times with carbon tetrachloride and then rinsing thoroughly in warm water containing a small amount of Deceresol OT. The Deceresol OT was added to insure complete wetting out of all parts of the fibers. Staples were carefully selected and placed in cotton mesh bags to hold the tips and bodies of the fiber in the same relative position as it would be found in the fleece. The dye bath was prepared with distilled water to which was added 10% Glauber's Salt, and 5% acetic acid 28%. The bath ratio was 60-1. Five grams of staple were usually employed.

Fleece dyed with 3% Calcochrome Violet B assumed a scarlet shade when no metal was added to the dye bath. The addition of 3 p.p.m. of iron caused the tips to be dyed brown with no appreciable change in the shade of the body portions.

Microscopical examination of cross sections of the tips showed that most of them were olive brown, some had brown rings and scarlet centers and an occasional fiber section was scarlet. The cross sections of the body parts of the fibers showed all to be bright scarlet. If the "ironed" wool was then top-chromed, there was little change in the shade of the tip portion of the staple but the body portion became violet.

Two per cent Calcochrome Alizarine Red SC dyed a staple a reasonably uniform brownish red shade, although the tips were slightly lighter in shade. When a similar dyeing was carried out using 1 p.p.m. of aluminum in the dye bath, the tips became a bright scarlet but there was little change in the shade of the body. Increasing the aluminum to 3 p.p.m. caused an unexpected result. The tips were now dyed a brighter, heavier scarlet, but the rest of the fleece was definitely weaker in shade. Another dyeing in which 5 p.p.m. of aluminum was used caused the scarlet tips to be dyed still more deeply, the depth of shade of the body portion being somewhat weaker than for either the 1 p.p.m. dyeing or the 3 p.p.m. dyeing.

TABLE 18

Concentration of Aluminum	*Color Strength of Body End*
0	100%
1 p.p.m.	90%
3 p.p.m.	78%
5 p.p.m.	60%

The procedure of the preceding dyeing was repeated, except that no aluminum was added to the dyebath until the acid shade had been obtained. Then 14.1 p.p.m. (equal to 0.75% aluminum sulfate containing 18 moles of water of crystallization) was added. A comparatively uniform scarlet shade resulted, indicating that if an excess of aluminum was used, both the tips and the body portion were metallized and were uniform in shade.

In order that the actual color values of the unmetallized body portions of the above dyeings be determined accurately, the color strengths as determined on the spectrophotometer are shown in Table 18.

It is obvious that preferential metallization of the dye in the tip portion is being obtained. The exact mechanism by which the addition of 3 p.p.m. or 5 p.p.m. of aluminum to a 2% Alizarine Red SC dyeing causes more intensely dyed tips and less strongly dyed body portions than obtained when 1 p.p.m. is used, is not at present known.

Preferential metallization experiments were repeated using Calcochrome Blue Black Conc. (C.I. 202). A staple dyed with this dye without metal gives a self-shade which is a dull, reddish brown. When 3 p.p.m. of copper are added to the dyebath, the tips become reddish violet. If this "coppered" dyeing is subsequently top-chromed, the body portion becomes dark blue but there is little change in the shade of the tip portion.

Dyeings were repeated using Calcochrome Violet B, but 5 p.p.m. of chromium were substituted for the iron used in the previous experiment. Again, the body was scarlet but the tips were an intense violet. When sufficient p.p.m. of chromium were used to completely chrome all of the dye, the tips and body parts were approximately the same shade of violet, and showed some contrast in depth of color between the tip and body portions.

Calcochrome Fast Green SGV (C.I. 292) was dyed both as a self-shade and with 1, 3, and 5 p.p.m. of cobalt. The self-shade of this color is a dull violet. The presence of cobalt in the bath caused the tips to become a bluish-grey. When these "cobalted" dyeings were top chromed, the tips remained predominantly bluish-gray and the body sections become predominantly green.

Similar dyeings were made using Calcochrome Brown RH and 3 p.p.m. of nickel. The body portion of the staple was dyed a rather uniform brownish orange, which is the shade of the unmetallized dye. However, the dyeing containing the 3 p.p.m. of nickel resulted in reddish brown tips. If this "nickeled" dyeing was subsequently top chromed, the body portion acquired the typical Brown RH shade, but due to the nickel complex, a high proportion of the tips retained the reddish brown shade.

In the preceding dyeings, qualitative results have been obtained which indicate that trace metals tend to concentrate in the tips of the staple and react with the dyestuff to give a metallized dye. These metallized dyes are fixed in the tips of the wool fibers, and in many cases the metals are not displaced if the dyed wool is subsequently chromed. Consequently, in practice, the middle and body portions of the fiber may be the desired chromed shade, but the tip portion, due to trace metals, may have a totally different and undesired shade. Such bicolored fibers give rise to heathery and skittery effects.

An unusual example of the dependence of tippy dyeing upon metals lodged in the tips of wool fibers was found when a portion of one of the Australian fleeces was dyed in distilled water with 6% Calcochrome Alizarine Red SC. Boiling was continued for 1 hour. Then the wool was rinsed thoroughly and dried. Examination of the dyed fleece showed that a number of the staples exhibited a bluish-red shade at the extreme ends of the tips, seeming to indicate that iron had been present in the dye bath. Since iron had not been added, tips and body portions of the undyed staples were analyzed to determine iron content colorimetrically. For comparison, a similar staple was "dyed" in a dyebath identical to that used above but without the addition of dye.

The presence of 0.06% iron in the tips at the start of the dyeing would be sufficient to produce the effects observed. It is interesting to note also that the tips of the wool fibers release iron and other inorganic materials when they are boiled in a blank dye bath without dye, as is shown in Table 19. However, when dye is present, it apparently penetrates the damaged tips and so combines with the iron in the early stages of the dyeing operation as to produce the iron shade. In order to con-

TABLE 19

	Fleece extracted with CCl_4 and rinsed in warm water		*Fleece boiled for 1 hour in a blank dyebath*	
	% Ash	*% Iron*	*% Ash*	*% Iron*
Tips	3.5%	0.06	1.10	0.02
Body	1.3%	0.01	0.92	0.01

firm the iron effect, a 2% dyeing of Calcochrome Alizarine Red SC was made with an addition of 3 p.p.m. of iron to the dyebath. All staples had dark violet tips similar in shade to those described a ove, and most of the body portions were slightly bluer in shade than the normal unchromed acid dyeing.

From the analytical results given above, it would appear that the iron content is more uniform throughout the fiber after the fiber has been boiled in an acidified bath. Tippiness in dyeing should, therefore, be reduced by the boiling treatment. This has been found to be the case. When boiled wool was dyed with Alizarine Red SC, the tips and body were the same shade, and none of the tips exhibited violet areas. Race *et al (8)* have stated that a greater iron content in the tips of wool may be a source of unlevel dyeing in the case of dyes that are sensitive to iron. The present experiments indicate that their assumptions are correct in that the presence of iron can change the shade in the tips.

The effect on tippiness of scouring with soaps, sulfate alcohols, and alkalies such as ammonia and soda ash was also investigated. Fleece was prepared for dyeing by (a) extracting with carbon tetachloride followed by warm water rinses, (b) scouring with 6% soap flakes and 3% soda ash for 20 minutes at 120° F. or (c) scouring with 3% sulfated alcohol and 5% ammonia for 20 minutes at 120° F. After several rinses in warm water, portions of each of these samples were dyed with 2% Calcochrome Alizarine Red SC and an addition of 3 p.p.m. of aluminum. Wool cleaned by all three treatments produced scarlet tips and bluish-red body parts. The wool scoured with sulfated alcohol and ammonia, however, dyed a more yellow body shade than did the wool treated by the other two methods.

Similar samples pre-treated as above were dyed with 2% Calcochrome Brown PG and 6% ammonium sulfate, and boiled 1 hour. In each case, the tips were practically undyed while the parts adjacent to the tips were heavily dyed. In general, the body areas were lightly dyed.

These experiments indicate that (a) the method used in cleaning the staple did not produce level dyeing, and that (b) boiling in an acid solution does something which ordinary soap or solvent cleaning does not do.

To confirm the dyeing results, spectrographic analyses were made of the ash of the unchromed tips and body portions of staples from each dyeing to which trace metals had been added. It was found that the tips contained a higher metal content than did the body portions of the fibers. It is believed that if all damaged areas and fiber tips could be completely separated prior to analysis, the body sections would be found to be practically free of trace metals when 1 p.p.m. or less is added to the dyebath.

Quantitative determinations on the ash and moisture content of several dyeings made with and without uranium gave the results shown in Table 20. These analyses provide rather conclusive proof that during the dyeing operation a considerable portion of the uranium metal used in these experiments was removed from the dyebath and concentrated in the tips and other damaged areas.

Radioaction Studies

Further proof of the concentration of metals in tips and in other damaged parts of the fiber was obtained by means of radioautographs. Certain dyes such as Calcochrome Alizarine Red SC, Calcochrome Blue Black Conc. and Calcochrome Brilliant Blue BBG form metal complexes with uranium salts. The uranized shade of Alizarine Red SC is a reddish blue, that of Blue Black Conc. is a dull violet, and that of Brilliant Blue BBG is a bright reddish blue. By boiling one hour and rinsing, dyeings were made on Australian fleece, using 6% of Alizarine Red SC, 10% calcined Glauber's salt, 8% acetic acid 28%, and 20 p.p.m. of uranium, as uranium acetate. The extreme tips of the fibers were black and the body was a dull crimson. Other 6% dyeings of Red SC were topped with 1, 2, 3, 4, 5, and 6% of uranium acetate. Examination of the fleece showed that all tips were reddish blue and that each increase in the amount of uranium salt used caused the body to be slightly bluer. Other dyeings of Alizarine Red SC and Brilliant Blue BBG were obtained by a meta-uranium (uranium acetate in the dye bath) method. All dyeings were rinsed thoroughly and air dried. Staples from each dyeing were then placed on Tri-X Panchromatic film and allowed to remain in contact for 70 days. The developed film showed that a radioautograph of the parts containing uranium had been obtained. When the amount of uranium acetate used was less than that required for complete metallization of all the dye present, the tip areas produced a much brighter image. When an excess of uranium salt was used, all parts of the fleece produced an image, but the tip areas always produced a greater effect upon the film.

The radioautographs are produced by a combination of the alpha rays from Uranium 238 and the beta and gamma rays from disintegration products of the uranium. These rays affect a photographic film and reveal differences in concentration of uranium by brighter and darker areas. In all cases the tip areas were brightest, showing that the tips contained a greater concentration of uranium than did other parts of the fleece. Figures 21 and 22 show staples of fleece dyed with an addition of uranium salt. Figures 23 and 24 are the radioautographs produced by these same staples.

Further experiments were made using radioactive cobalt isotope (Co^{60}) prepared at Oak Ridge by means of neutron pile bombardment. Co^{60} is both a beta and gamma emitter. The energy of the beta rays is 0.3 million electron volts (rather weak) and that of the gamma rays is 1.1, 1.3 million electron volts (comparatively strong). Because the alpha rays emitted from the uranium used in the previous

TABLE 20
Australian Fleece

	Undyed fleece extracted with CCl_4—rinsed with water		*Undyed fleece Boiled 1¼ hrs. with 10% Glauber's 8% acetic acid 28%*		*Dyed fleece 6% Aliz. Red. SC 10% Glauber's 8% acetic acid 28% boil 1½ hours*		*Dyed fleece 6% Aliz. Red SC 8% acetic 28% 10% Glauber's 20 p.p.m. Uranium boil 1¼ hours*		*Dyed fleece 6% Aliz. Red SC 8% Ammonium Sulfate 6% Uranium acetate boil 30 min.—add 3% acetic acid 28% boil 45 minutes*	
	Tip	*Body*	*Tip*	*Body*	*Tip*	*Body*	*Tip*	*Body*	*Tip*	*Body*
Percent Ash	3.30	1.01	1.46	1.12	1.45	0.99	1.78	0.91	4.72	1.83
Percent Moisture Lost in 1 hour at 100°C...	10.9%	10.7%	10.6%	10.7%	10.8%	10.7%	9.8%	10.7%	10.4%	10.6%

experiment have a very low penetrating power and only very small quantities of the beta gamma emitting products are formed, long periods of exposure were required to produce an autograph. With artificially active beta and gamma emitting isotopes, the amount of activity taken can be varied by regulating the proportions of the active isotope and the inactive "carrier" which are taken. It was estimated that about 5% of radiocobalt chloride in 95% "carrier" cobalt acetate would produce enough activity on a staple of wool to get an autograph in about 24 hours.

Two per cent dyeings of Calcochrome Brown PG, Brown RV, Green SGV and Brown RH were made, using 2 and 4 p.p.m. of the cobalt chloride acetate mixture, 10% Glauber's Salt, 6% ammonium sulfate, liquor ratio 1-60. Dyeings were brought to the boil in 30 minutes. They were then boiled one hour, rinsed thoroughly, and air dried. Visual examination of the dyed staples showed that the tips were the cobalt shade of each dye, while the middle and body parts were the unmetallized shade.

The autographs were made on Eastman "no-screen" X-ray film. A sheet of 0.001 of Cellophane was placed between the wool and the film, and the three were pressed together between steel plates held by "C" clamps. Examination of the radioautographs showed that there had been

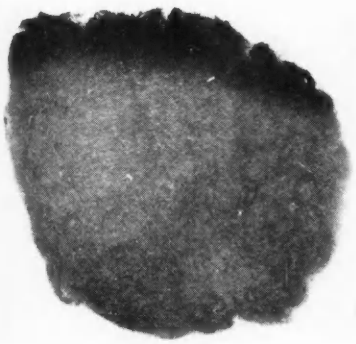

FIGURE 21
Photograph of a staple dyed with
6% Calcochrome Alizarine Red SC
10% Calcined Glauber's Salt
10% Acetic Acid 28%
2% Uranium Acetate
Boiled 1 hour

FIGURE 22
Photograph of a staple dyed with
6% Calcochrome Brilliant Blue BBG
10% Calcined Glauber's Salt
10% Acetic Acid 28%
6% Uranium Acetate
Boiled 1 hour

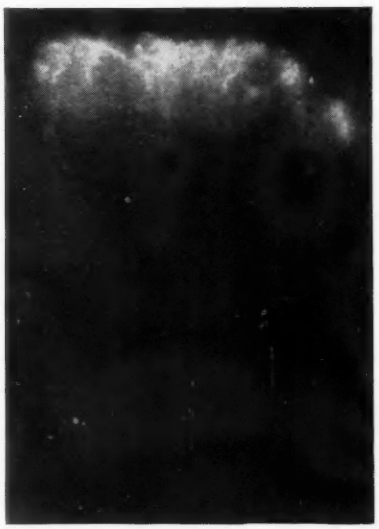

FIGURE 23
Radioautograph of a staple dyed with
6% Calcochrome Alizarine Red SC
10% Calcined Glauber's Salt
10% Acetic Acid 28%
6% Uranium Acetate
Boiled 1 hour
In contact with film 70 days.

FIGURE 24
Radioautograph of a staple dyed with
6% Calcochrome Brilliant Blue BBG
10% Calcined Glauber's Salt
10% Acetic Acid 28%
6% Uranium Acetate
Boiled 1 hour
In contact with film 70 days.

FIGURE 25
Radioautograph of a staple dyed with
2% Calcochrome Fast Green SGV
10% Calcium Glauber's Salt
6% Ammonium Sulfate
2 p.p.m. Radiocobalt chloride-acetate mixture
Boiled 1 hour
In contact with film 24 hours.

FIGURE 26
Radioautograph of a staple dyed with
2% Calcochrome Fast Green SGV
10% Calcium Glauber's Salt
6% Ammonium Sulfate
2 p.p.m. Radiocobalt chloride-acetate mixture
Boiled 1 hour
In contact with film 24 hours.

FIGURE 27

Radioautograph of a staple dyed with 4 p.p.m. Radiocobalt chloride-acetate mixture Soaked in "blank" dye bath for 24 hours at 80°F.
In contact with film 24 hours.

preferential absorption of the cobalt by the tips of the fibers. (Figures 25-26.) There was also a significant amount of cobalt absorbed by the remainder of the fiber.

Three samples of the fleece wool were treated with 4 p.p.m. of cobalt, with the same amounts of Glauber's salt and ammonium sulfate which were used in the dyeings, but no dye was present. These samples were soaked in this solution for 24 hours at approximately 80° F. (27° C.)

The autograph for the sample treated with cobalt, but without any dye, was similar to that of the dyed samples, showing that cobalt was absorbed in a comparable manner both with and without the dye. (Figure 27.)

RADIOACTIVE COUNT. — Samples were cut from various portions of each of the dyed staples of wool to determine the amount of cobalt absorbed. One sample, which consisted as nearly as possible of material which was dyed to a different color from that of the major portion of the wool, was taken from the tip portion of each staple. A second sample was taken from a region near the tip, but consisted as nearly as possible of material which did not show the characteristic cobalt-complex color of the tip. Other samples were taken from the middle and from the root end of the fiber. Finally a portion of the full length of the fiber was used. The amount of radioactive cobalt absorbed on the various portions of wool was determined by Geiger counter measurements. Each sample was weighed and the activity per unit weight was obtained (counts/minute/mg. wool). The resulting values are shown in Table 28. These values have been corrected for the self-absorption of the radiation within the sample.

The ratio of the activity in a given portion relative to that in the tip portion of the same sample is shown in Table 29.

It appears from the activity measurements shown in Table 29 that there was a general increase in the amount of cobalt absorbed along the length of the fiber from the root end through the middle to the tip end. The most marked change was encountered between the tip and middle portions. The near-tip portion in some cases had almost as much absorbed cobalt as the tip portion, but in other cases it had only a little more absorbed cobalt than the middle portion. These variations in the cobalt content of near-tip regions probably are caused by the extent of damage in each portion. These radiocobalt studies have illustrated a relatively simple technique for obtaining quantitative information about the absorption of metals or metallized dyes by wool fibers.

Other natural or artificially prepared radioactive isotopes of elements such as thorium, aluminum, chromium, cobalt, copper, iron, nickel, etc., can be used as tracers providing they form metal complexes with the dye selected for study.

TABLE 28

Activity Measured on the Wool

Dye	Cobalt p.p.m.	Temp. °C.	Corrected activity (C./m./mg.wool) Tip	Near Tip	Middle	Root	Full Length
Brown PG	2	100	580	420	115	...	...
Brown PG	4	100	740	580	260	200–	235
Brown RV	2	100	530	310	140	80	160
Brown RV	4	100	1020	930	380	130	240
Brown RH	2	100	710	120	40	36	89
Green SGV	2	100	750	230	200	210a	370
none	4	27	760	160	160	90	180

Note: a. Sample taken from inside of middle portion.

TABLE 29

Relative Activity Compared to Tip

Dye	Cobalt p.p.m.	Temp. °C.	Activity/Activity in Tip Near Tip	Middle	Root	Full Length
Brown PG	2	100	0.72	0.19	...	...
Brown PG	4	100	0.78	0.24	0.27	0.32
Brown RV	2	100	0.58	0.26	0.15	0.30
Brown RV	4	100	0.91	0.37	0.13	0.24
Brown RH	2	100	0.17	0.06	0.05	0.13
Green SGV	2	100	0.31	0.27	0.28	0.49
none	4	27	0.21	0.21	0.12	0.24

TABLE 30

Dyed	Unchromed Acid Dyeing	Top Chromed in Exhausted Dye Bath	Top Chromed In Fresh Bath
Calcochrome Orange LC	Tips lightly dyed	Tips heavier	Tips lighter
Calcochrome Red ECB	Tips lightly dyed	Tips heavier	Tips lighter
Calcochrome Aliz. Red SC	Tips lightly dyed	Tips heavier	Tips lighter
Calcochrome Violet B	Tips lightly dyed	Tips lighter	Tips lighter
Calcochrome Brill. Blue BBG	Tips lightly dyed	Tips lighter	Tips lighter
Calcochrome Cyanine RC	Tips lightly dyed	Tips heavier	Tips lighter
Calcochrome Blue Black Conc.	Tips lightly dyed	Tips heavier	Tips lighter
Calcochrome Fast Green SGV	Tips lightly dyed	Tips lighter	Tips lighter
Calcochrome Brown PG	Tips lightly dyed	Tips heavier	Tips lighter
Calcochrome Brown RH	Tips and body similar	Tips heavier	Tips and body similar
Calcochrome Brown EB	Tips and body similar	Tips heavier	Tips heavier

Tippy Effects Caused by Top Chroming

It was observed that top chroming in the dyebath can cause positive tippy effects with a number of dyes. To investigate this effect, a series of tests was run with 11 chrome dyes. Three dyeings of each were made on Australian fleece, using 2% of dye, 10% Glauber's salt, 5% acetic acid 28%, and boiling for 45 minutes. In dyeing No. 1, the fleece was removed, rinsed, and dried without top chroming. Dyeing No. 2 was top-chromed by adding 1% sodium bichromate to the "exhausted" dye bath (single bath process) while dyeing No. 3 was top-chromed in a fresh bath, using 1% chrome and 2% acetic acid 28% (two-bath process). A fourth dyeing was

obtained by adding undyed fleece to the "exhausted" dye bath (saved from dyeing No. 1), and adding 1% bichromate. These were boiled 40 minutes, rinsed, and dried. The results are shown in Table 30.

It was observed that practically all the dyeings chromed in a fresh bath lose considerable color value as compared with dyeings chromed in the exhausted dye bath. In the latter case, the dyeing generally exhibits one of two tip effects: (A) a change from undyed or lightly dyed to more heavily dyed tips as shown by eight of the eleven dyes; or (B) the tips may remain light as in the case of Green SGV. The heavy tip effect is probably caused by the fact that the "exhausted" bath contains from 5 to 20% of the original dye. In effect, the addition of bichromate to a bath containing an appreciable amount of dye converts it to an acid metachrome process, in which case most of the dye remaining in the "exhausted" bath is deposited in the tips. With the exception of Brilliant Blue BBG, all of the dyes used in this experiment exhibited prominent tippy dyeing when they were applied by the metachrome procedure *(7)*.

The light tips of the Green SGV probably resulted from the fact that the dye bath had been completely exhausted before the chrome was added. Confirmation of this was obtained by adding 0.25% of Green SGV to the exhausted dye bath five minutes before the addition of the bichromate. Before this dye was added the tips had been light; after a further boiling of 30 minutes, the tips became much darker than the body. When chromed in a fresh bath containing acid and bichromate, those dyes which have strong affinity for the wool do not bleed off but remain where they are absorbed originally, and light tips result. Dyes such as Brown EB, however, bleed into the fresh bath during the early stages of the chroming process, and the presence of chrome in the bath causes dye to be reabsorbed and concentrated in the tips, thereby producing positive effects.

In the foregoing experiments, and in many others not described in detail, the metallized dye is concentrated in the tips and other damaged areas, almost to the exclusion of other parts of the fiber, when limited quantities of metal are present. This concentration of metallized dye in the tips may occur by two mechanisms. The metal salt in the dye bath may be absorbed rapidly by the tips and damaged areas, held by the fiber, and subsequently react with the dye to form the metal-dye complex. In this mechanism, the metal salt is first absorbed by the fiber and then the metal-dye complex is formed. On the other hand, it is possible that the dye and soluble metal salt combine in the dye bath to give the metal-dye complex, which is then preferentially absorbed by the tips and damaged areas *(9)*. It was found that a few chrome dyes will not dye tippy even though 3 p.p.m. of aluminum, cobalt, iron, copper, or nickel, are added to the dye bath at the start. Copper showed much less tendency to cause tippy dyeing than did other metals studied, although it does cause definite changes in both the chromed and unchromed shades.

Effect of Metal Equipment

Many dyeing machines still in use have linings or other parts made of aluminum, copper, iron or Monel metal. The dyeing procedure in an acid bath, in metallic equipment, may convert traces of the metal to a soluble compound which reacts with the dye to form a metal complex. This effect is more pronounced when dyeing by the top chrome method, because the reaction between impurity metals and dye is completed before addition of the bichromate. Experiments have shown that drastic alterations in shade are obtained when sheets of metal placed in the dye bath are substituted for p.p.m. of metal added as soluble salts. In general the addition of iron sheets alters the shade, and the effect is roughly proportional to the surface area of the sheet exposed to the dye bath. One per cent dyeings were made using 10% Glauber's salt, 4% acetic acid 28% at a liquor ratio of 60:1 Dyeings were boiled for 1 hour. No metal was added to one dyeing, another was topped with 0.75% ferrous sulfate, others were dyed in the presence of 12.5 cm.^{2}, 25.0 cm.^{2},37.5 cm.^{2}, and 50 cm.^{2} sheets of metallic iron, copper and aluminum. These dyeing were then measured for per cent metallization by the method developed by E. I. Stearns *(12)* on the spectrophotometer. The dyeings without metal were used as a standard for zero metallization, and the dyeings topped with the metal salts were used as the standard for 100% metallization. Different dyes were used with the various metals depending upon the extent of the shade change during metallization. The results are given in Table 31.

These analyses indicate the extent to which chrome dyes can be affected by the use of certain metals in dyeing machinery.

Copper and aluminum are sometimes used for linings and other dyeing machine parts, and so their effect upon the shades of Calcochrome Blue Black Conc. and Alizarine Red SC was studied as described above. It was observed that copper and aluminum metals, unlike the iron, released fewer metal ions into the dye bath, and therefore the tips contained most of the metallized dye. A small amount of these metals was absorbed by the middle and body parts also.

TABLE 31

Area of Metal Present in 300 ml. of dyebath	*Approximate Percent of Dye Metallized*		
	*Iron**	*Copper***	*Aluminum****
12.5 cm.^{2}	73%	21%	6%
25.0 cm.^{2}	93%	24%	10%
37.5 cm.^{2}	97%	30%	16%
50.0 cm.^{2}	99%	42%	17%

* Calcochrome Fast Red ECB.
** Calcochrome Blue Black Conc.
*** Calcochrome Alizarine Red SC.

Bottom Chrome Method

A study of the chrome bottom method of dyeing provided further proof of preferential metal absorption by tips and other damaged areas in wool fibers and fleece. Several series of experiments were run by bottom chroming Australian and South American BA's wool fleece, with concentrations of sodium bichromate starting with 0.1% bichromate on the weight of the wool and increasing by tenths up to 2%. Two to four per cent of cream of tartar was used, and the wool was boiled for one to one and one-half hours, depending upon the amount of bichromate used. The chrome-bottomed wool was then dyed with dyes which have contrasting acid and chromed shades.

Australian fleece, chrome bottomed as above, was dyed in a fresh bath with 1% Calcochrome Brilliant Blue BBG and 5% ammonium sulfate. After it had boiled 30 minutes, an addition of 2% acetic acid 28% was made, then the boiling was continued for 30 minutes longer. Examination of the dyeings showed that although the wool treated with 0.1% and 0.2% bichromate had brilliant blue tips, the remainder of the fleece was a brown (unchromed) shade. (Figure 32.) Dyeings on wool bottom-chromed with 0.5% and 1.0% bichromate showed the former to be quite uniform in shade from tip to body, whereas the latter had tips which were lighter and greener than was the average of the body fibers. (Figure 33.)

When the wool was bottom-chromed with 0.1% and 0.2% of bichromate, dyeings of Calcochrome Orange LC, Red ECB, Red SC, Violet B, Green SGV, and many others had tips the color of the chromed shade, while the middle and body parts were the unchromed shade. Occasional exceptions to this rule may be

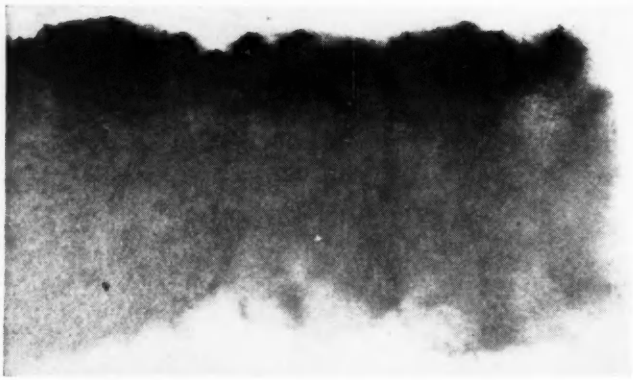

FIGURE 32
Staples bottom chromed with 0.2% bichromate
Dyed with
1% Calcochrome Brilliant Blue BBG
5% Ammonium Sulfate
Boiled 30 minutes, add
2% Acetic Acid 28%
Boiled 30 minutes

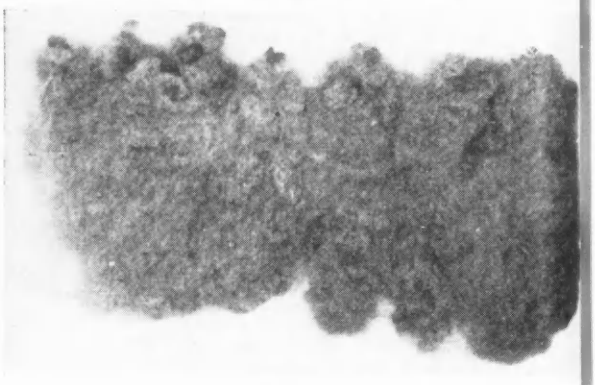

FIGURE 33
Staples bottom chromed with 1.0% bichromate
Dyed with
1% Calcochrome Brilliant Blue BBG
5% Ammonium Sulfate
Boiled 30 minutes, add
2% Acetic Acid 28%

found with dyes which do not exhibit tippiness by the top and meta chrome procedures in the presence of chrome or other metal. These dyes do not dye tippy on chrome-bottomed wool.

Fleece wool chrome bottomed with 0.1%, 0.2%, 0.5%, and 1% bichromate and 4% cream of tartar was examined prior to dyeing, and distinct differences in shade between the tip and body were noted. The 0.1% bichromate wool had bright yellowish-orange tips and white body, 0.2% bichromate produced wool the tips of which were heavier and slightly browner, and the body portions had a faintly greenish cast. Wool bottomed with 0.5% bichromate had tips similar in shade to the 0.2% bichromate treatment, but the body portions were distinctly green; the wool treated with 1.0% bichromate had tips similar in color to that produced with 0.5% bichromate, but the middle and body sections were bluish green. These studies indicate that the chrome deposited in the tips is in a different state from that absorbed by the middle and body areas.

Australian fleece treated in the same manner as described previously for the chrome bottom studies was boiled with 4% cream of tartar and 0.1%, 0.2%, 0.5%, and 1.0% of ferrous sulfate, copper sulfate, aluminum sulfate, nickel sulfate, and cobalt acetate. The wool was then rinsed thoroughly and dyed with 1% Calcochrome Violet B, 5% ammonium sulfate. It was boiled for 30 minutes, an addition of 2% acetic acid 28% was made, and then it was boiled for 30 minutes longer. Examination of staples from each dyeing showed that, although none of the metals produced such distinct contrasts in shade or extremes in tippiness as were obtained with the bichromate, nevertheless the tips and adjacent areas were somewhat darker than the body portions.

From these and other experiments, it can be concluded that the tips of the fibers absorb chromium (and other metals) first. Then the other parts of the fibers absorb it in proportion to the amount of chrome available. This preferential absorption is associated with tippy dyeing in bottom chrome wool, and accounts to some extent for the variations in shade and penetration noted previously *(11)* when cross sections of bottom-chromed dyeings were examined microscopically. Unequal absorption and fixation of chrome between tips and body parts during the bottom-chrome process may account also for the lack of fastness exhibited by certain chrome shades during wet finishing operations. If a high proportion of the chrome is absorbed by the tips and damaged areas, too little chrome may be deposited in the body areas. During dyeing, the color is absorbed first as the unchromed dye and then it combines with the chrome in the fiber *(9)*. If there is insufficient chrome present in the body areas to completely metallize all of the absorbed dye, some of it will remain present as the acid dye in the finished shade. Bleeding of color and staining of other materials will be proportional to the amount of unchromed dye in the fibers. When maximum fastness is required, bottom chromed fabrics are sometimes top chromed to improve the fastness. Complete chromation of the dyes is thus assured.

Metachrome Dyeing

There is a direct relationship between tippy dyeing and the method of application of the dye. It has already been observed (7) that Acid Anthracene Brown LE (Pr. 204) produces lighter tips by the top chrome procedure and heavier tips by the metachrome procedure. During the present investigation it was observed that many dyes produced tippy effects by the standard metachrome process, although they produced uniformly colored dyeings when they were applied top chrome. This indicated that the presence of metal, e.g. chromium, was one of the direct causes of tippy dyeing.

2% dyeings of Calcochrome Violet B, Brown RH, Red EBC, Alizarine Red SC, Brown PG, Cyanine RC and Chrome Brown LE were dyed with 5% ammonium sulfate, both with and without 1% sodium chromate. Dyeings were boiled for one hour, then 2% acetic acid 28% was added, and the boiling was continued for 30 minutes. All the dyes gave very heavy tips (positive) except the Brown LE, which produced a medium heavy tip. When the sodium chromate was omitted, Brown RH, Cyanine RC, and Violet B were uniform in shade from tip to body, but Red ECB, Red SC, Brown PG and Brown LE gave negative tippiness. Since the only variable between the two sets of dyeings was the chrome, it was concluded that the action of this metal caused the tips to dye darker.

Calcochrome Violet B was dyed by the meta process, substituting iron, copper, nickel, aluminum, and cobalt salts for the chromium. Five p.p.m. of each metal was used. These experiments showed that under the conditions of the test, the presence of iron and copper salts in the dyebath changed the shade slightly but did not cause the tips to dye heavier. The dyeing made with aluminum changed the

TABLE 34

1% Calcochrome Dye	C.I.	Shades					Metal Sensitivity Visual		
		No Metal	Top Chromed 0.75% Bichromate	Topped with 0.75% Ferrous Sulfate	Topped with 0.75% Copper Sulfate	Topped with 0.75% Aluminum Sulfate	Iron PPM	Copper PPM	Aluminum PPM
Calcochrome Yellow 3G Conc.	52	light yellow	greenish yellow	light brownish yellow	light greenish yellow	deeper than no metal	1	1	−1
Calcochrome Yellow EFA Conc.	219	light yellow	deep greenish yellow	light brownish yellow	light greenish yellow	deeper than no metal	1	1	1
Calcochrome Yellow G	Pr. 56	light reddish yellow	deep greenish yellow	light brownish yellow	light greenish yellow	deeper than no metal	1	1	−1
Calcochrome Yellow 2G	36	light reddish yellow	deep yellow	medium brownish yellow ;	light reddish yellow	much heavier than no metal	1	1–2	1
Calcochrome Yellow CGW 250%	343	deep golden yellow	deep reddish yellow	deep brownish yellow	deep yellow	deeper than no metal	1	1	−1
Calcochrome Yellow FC	Pr. 1	medium canary yellow	deep reddish yellow	medium greenish yellow	greenish yellow	slightly deeper than no metal	1	1	1
Calcochrome Orange GR	274	deep brownish yellow	deep reddish orange	light brownish orange	dull yellowish orange	deep bright orange	1	1	1
Calcochrome Orange LC	—	medium reddish orange	medium reddish orange	yellowish olive brown	dull brownish yellow	deeper bright orange	−1	−1	1
Calcochrome Orange R Ex. Conc.	40	medium reddish yellow	deep reddish orange	medium brownish orange	dull brownish yellow	deep bright orange	1	1	1
Calcochrome Brown EB	Pr. 14	medium reddish brown	deep brown	medium brown	medium brown	yellowish brown	1	1	1
Calcochrome Brown EBR	Pr. 14	medium reddish brown	deep brown	deep yellowish brown	medium reddish brown	slightly yellower than no metals	1	−1	1
Calcochrome Brown PG	Pr. 4	light reddish brown	medium brown	medium yellowish brown	medium brown	on tone slightly heavier	1	1	1
Calcochrome Brown RH	98	medium orange brown	deep brown	medium orange brown	medium reddish brown	heavier, much brighter yellower	−1	−1	−1
Calcochrome Brown RV Conc.	Pr. 2	medium reddish brown	medium violet-brown	medium yellowish brown	fuchsia	deep rich fuchsia	−1	−1	−1
Calcochrome Fast Red ECB	652	medium orange	bright bluish red	medium yellowish brown	medium yellowish red	medium coral	−1	1–2	1
Calcochrome Alizarine Red SC	1034	brownish red	yellowish red	dull violet	bluish red	bright scarlet	−1	1	−1
Calcochrome Silk White Red B	216	medium coral	medium bluish red	light reddish tan	light yellowish tan	yellower and brighter	1	1–2	1–2
Calcochrome Violet B	169	medium scarlet	deep dull violet	deep olive brown	fuchsia	medium bright fuchsia			
Calcochrome Brilliant Blue BBG	720	light brown	brilliant blue	very dull blue	very dull reddish violet	medium bright blue	1	1–2	1–2
Calcochrome Cyanine RC	722	very light pink	medium bright blue	thin dull greyish blue	dull reddish violet	medium bluish violet	1	1	1
Calcochrome Fast Blue BG	Pr. 93	medium bright reddish violet	dull greenish blue	medium brown	medium bluish violet	bluish violet	−1	1	−1
Calcochrome Alizarine Blue Black B	1085	reddish dull violet	reddish grey	dull bluish violet	dull brownish violet	bluer than no metals	1	1–2	1–2
Calcochrome Alizarine Blue Black 3B	1085	dull bluish violet	greenish grey	dull violet	dull brownish	much bluer than no metal	2–3	1	1
Calcochrome Blue Black Conc.	202	dull reddish brown	medium blue	reddish brown	bright reddish violet	medium bright bluish violet	−1	−1	−1
Calcochrome Blue Black BC	201	dull violet	greenish blue	medium brown	bright bluish violet	medium bright blue	−1	−1	1
Calcochrome Black T Ex. Conc.	203	brownish violet	dull grey	medium brown	reddish violet	medium dull blue	−1	−1	−1
Calcochrome Black F Conc.	299	reddish blue	deep grey	dull blue	dull blue	medium dull blue	1–2	1	1–2
Calcochrome Black FB Conc.	299	reddish blue	deep grey	dull blue	dull blue	medium dull blue brighter than Black F	1–2	1	1
Calcochrome Black AC	204	dull violet	light grey	light brown	medium violet	light dull violet	−1	−1	1
Calcochrome Black PV Conc.	170	medium reddish violet	dull blue	medium brown	medium bluish violet	medium dull reddish violet	−1	−1	1

TABLE 35
Wool Wash Test #3
Staining

Shade Change	Cotton	Rayon	Bemberg	Nylon	Acetate	Wool	Fading observed in 32-hour exposure in Fadeometer
1% Calcochrome Orange LC							
No metal 2	4	4–5	4–5	5	5	3	color destroyed
Aluminum 2	4	4–5	4–5	5	5	3	distinct
Chrome 4–5	4–5	4–5	4–5	5	5	4–5	none
Copper 3	4–5	4–5	4–5	5	5	3–4	none
Iron 3	4–5	4–5	4–5	4	5	4	very slight
1% Calcochrome Red ECB							
No metal 3	4	4	4	5	5	3–4	considerable
Aluminum 2	4	4	4–5	5	5	3–4	considerable
Chrome 4–5	5	5	5	5	5	5	slight
Copper 4–5	3–4	3–4	3–4	4–5	5	3	slight
Iron 3–4	4–5	4–5	5	5	5	4	distinct
1% Calcochrome Alizarine Red SC							
No metal 2	4–5	4–5	5	5	5	2–3	color destroyed
Aluminum 3	4–5	4–5	4–5	5	5	3	none
Chrome 4–5	5	5	5	5	5	5	very slight
Copper 4	3	3	3	5	5	3–4	very slight
Iron 3	5	5	5	5	5	3–4	slight
1% Calcochrome Violet B							
No metal 2	4–5	4–5	5	5	5	2–3	considerable
Aluminum 2	4–5	4	5	5	5	3	distinct
Chrome 4	5	5	5	5	5	4–5	none
Copper 4–5	4	4	4–5	4–5	5	1–2	none
Iron 4	5	5	5	5	5	4	slight
1% Calcochrome Brown EB							
No metal 3	2	2	2	4	4	2	considerable
Aluminum 3	2	2	2	4	4	2	considerable
Chrome 5	4	4	4	5	5	4–5	very slight
Copper 5	3–4	3–4	3–4	4–5	5	4–5	slight
Iron 5	3–4	3–4	3–4	4–5	5	4–5	distinct
1% Calcochrome Brown PG							
No metal 2	4–5	4–5	5	5	5	3–4	distinct
Aluminum 2	4	4	4	5	5	3–4	distinct
Chrome 4–5	5	5	5	5	5	5	slight
Copper 4–5	4–5	4–5	5	5	5	3–4	none
Iron 3	4–5	4–5	4–5	5	5	4	distinct
1% Calcochrome Brown RH							
No metal 2	4–5	5	5	5	5	4	considerable
Aluminum 2	4–5	5	5	5	5	4	considerable
Chrome 5	5	5	5	5	5	4–5	none
Copper 5	3–4	3–4	3–4	5	5	3	very slight
Iron 4	4–5	5	5	5	5	4–5	slight
1% Calcochrome Brown RV Conc.							
No metal 2–3	4	4–5	4–5	5	5	2–3	considerabe
Aluminum 3	4	4–5	5	5	5	2–3	very slight
Chrome 5	5	5	5	5	5	4–5	none
Copper 5	4–5	4–5	5	5	5	2–3	none
Iron 4	4–5	4–5	5	5	5	3–4	slight
1% Calcochrome Fast Green SGV							
No metal 2	2–3	2–3	2–3	5	5	3	considerable
Aluminum 2–3	2–3	2–3	2–3	5	5	3	considerable
Chrome 5	5	5	5	5	5	5	slight
Copper 3	4–5	4–5	4–5	5	5	4–5	distinct
Iron 3–4	4–5	4–5	4–5	5	5	5	considerable
1% Calcochrome Fast Blue BG							
No metal 2	5	5	5	5	5	4–5	considerable
Aluminum 2	5	5	5	5	5	4–5	considerable
Chrome 4–5	5	5	5	5	5	5	none
Copper 3–4	4–5	4–5	5	5	5	4–5	none
Iron 3–4	5	5	5	5	5	5	distinct
1% Calcochrome Brilliant Blue BBG							
No metal 1	5	5	5	5	5	4–5	color destroyed
Aluminum 1	5	5	5	5	5	4–5	considerable
Chrome 4–5	5	5	5	5	5	4–5	slight
Copper 1	5	5	5	5	5	4–5	color destroyed
Iron 1	5	5	5	5	5	4–5	considerable
1% Calcochrome Cyanine RC							
No metal 1	5	5	5	5	5	5	considerable
Aluminum 1	5	5	5	5	5	5	considerable
Chrome 4	5	5	5	5	5	5	slight
Copper 1	5	5	5	5	5	5	considerable
Iron 1	5	5	5	5	5	5	considerable
1% Calcochrome Blue Black BC							
No metal 2	4	4	4	5	5	2–3	considerable
Aluminum 3	4	4	4–5	5	4–5	3	distinct
Chrome 5	5	5	5	5	5	5	slight
Copper 4	4	4	4	5	4–5	2–3	none
Iron 3–4	5	5	5	5	5	4–5	distinct
1% Calcochrome Blue Black Conc.							
No metal 2	3–4	3–4	3–4	5	4	2–3	considerable
Aluminum 3	4	4	4–5	5	5	3	considerable
Chrome 5	5	5	5	5	5	5	very slight
Copper 4–5	3–4	3–4	3–4	4–5	5	2	very slight
Iron 4	5	5	5	5	5	3–4	distinct
1% Calcochrome Black T Extra Conc.							
No metal 2	3	3	3	4–5	5	2–3	considerable
Aluminum 3–4	4	3–4	3–4	4–5	3–4	3	considerable
Chrome 4–5	5	5	5	5	5	4–5	none
Copper 4–5	4	4–5	4–5	5	4	3–4	none
Iron 4	5	5	5	5	5	4–5	distinct

shade to a rich fuchsia, and the tips and body parts were similar. The use of aluminum sulfate as an equalizer of chrome dyes has been reported previously *(10)*. Chromium and cobalt both produced tippy dyeings in which the tips were heavier and different in shade from other parts of the fleece. Thus the presence in the dye bath of small amounts of soluble salts of chromium and cobalt causes preferential absorption and metallization of dye in the tips and damaged areas of the fibers.

When Calcochrome Red ECB was dyed by the meta method without chromium in the dye bath, the resultant dyeings were negative, that is, the tips were lighter than the body. If 5 p.p.m. of chromium were added at the beginning, the resultant dyeing was positive; that is, the tips were heavier than the body.

When Calcochrome Blue Black Conc. is dyed by the meta procedure without chromium in the dye bath, the fleece is well-dyed and the damaged areas are somewhat heavier. When bichromate is added at the beginning of the operation, only the tips are heavily dyed; the middle and body parts remain practically undyed. Apparently, such dyes as Calcochrome Fast Blue BG, Blue Black Conc., Blue Black BC, Black T, Black AC and Black PV are affected by the chrome and the wool during metachrome dyeing. The shade of the chromed dye formed in the tips quickly, and, as suggested by Race *et al (8)*, it may be that the chrome in the tips is not in the same state as is that absorbed by other parts of the fiber.

A few chrome dyes were found which did not dye heavily on the tips, regardless of the type of metal or variation in the metachrome method. However, the presence of chrome did affect the final result, because dyeings produced by the meta method without chrome invariably had tips which were lighter in shade than the body. When chrome was present, the tips were generally equal or slightly heavier in shade than the body.

Effect of Leveling Agents

In general, the adverse tippy effects caused by the presence of metals in the dyebath can be reduced or eliminated by leveling agents such as Calcomet Salt Powder. These agents, however, do not work equally well in all cases, because their effectiveness varies both with the dye and with the method of application. Various results are also obtained with the same dye, depending upon the metal or metals present. Experiments have shown a good top or metachrome leveling agent is one that will produce level dyeings when the agent is added at the beginning. It will also improve tippy dyeing which has

TABLE 36

AATCC Wool Wash Test #3

Staining

	Shade Change	Cotton	Rayon	Silk	Nylon	Acetate	Wool
1% Calcochrome Fast Red ECB							
Top chromed no impurity metals	5	5	5	4–5	5	5	5
aluminum 2 p.p.m.	5	5	5	4–5	5	5	5
cobalt 2 p.p.m.	4–5	5	5	4–5	5	5	5
copper 2 p.p.m.	5	5	5	4–5	5	5	5
iron 2 p.p.m.	4–5	5	5	4–5	5	5	5
nickel 2 p.p.m.	5	5	5	4–5	5	5	5
POTTING							
Top chromed no impurity metals	5–	5–	5	5*	5	5	5
aluminum 2 p.p.m.	5–	5	5	5*	5–	5	4–5
cobalt 2 p.p.m.	3–4	5	5	5*	5	5	5–
copper 2 p.p.m.	5–	5	5	5*	5	5	5–
iron 2 p.p.m.	3–4	5	5	5*	5–	5	5–
nickel 2 p.p.m.	5–	5	5	5	5–	5	4–5

FADEOMETER HOURS	20	50	100
Top chromed no impurity metals		88%	80%
aluminum 2 p.p.m.		85%	76%
cobalt 2 p.p.m.		92%	87%
copper 2 p.p.m.		85%	76%
iron 2 p.p.m.		80%	72%
nickel 2 p.p.m.		88%	80%

* staining on silk very slight reddish tint.

1% Calcochrome Alizarine Red SC							
Top chromed no impurity metals	3–4	5	5	5	5	5	5
aluminum 2 p.p.m.	3	5	5	5	5	5	5–
cobalt 2 p.p.m.	4	5	5	5	5	5	5
copper 2 p.p.m.	3–4	5	5	5	5	5	5
iron 2 p.p.m.	5	5	5	5	5	5	5
nickel 2 p.p.m.	4	5	5	5	5	5	5
POTTING							
Top chromed no impurity metals	3–4	5	5	5	5	5	4–5
aluminum 2 p.p.m.	3	5	5	5	5	5	4
cobalt 2 p.p.m.	4	5	5	5	5	5	4–5
copper 2 pp.m	3–4	5	5	5	5	5	4–5
iron 2 p.p.m.	5	5	5	5	5	5	4–5
nickel 2 p.p.m	4	5	5	5	5	5	4–5

FADEOMETER HOURS	20	50	100
Top chromed no impurity metals		100%	95%
aluminum 2 p.p.m.		100%	95%
cobalt 2 p.p.m.		96%	95%
copper 2 p.p.m.		100%	95%
iron 2 p.p.m.		98%	95%
nickel 2 p.p.m.		100%	95%

1% Calcochrome Brown RH							
Top chromed no impurity metals	4	5	5	5	5	5	5
aluminum 2 p.p.m.	4	5	5	5	5	5	5
cobalt 2 p.p.m.	4	5	5	5	5	5	5
copper 2 p.p.m.	4	5	5	5	5	5	5
iron 2 p.p.m.	4	5	5	5	5	5	5
nickel 2 p.p.m.	3–4	5	5	5	5	5	5
POTTING							
Top chromed no impurity metals	4	5	5	5	4–5	5	4
aluminum 2 p.p.m.	3–4	5	5	5	4–5	5	4
cobalt 2 p.p.m.	3–4	5	5	5	4–5	5	4
copper 2 p.p.m.	3	5	5	5	4	5	4
iron 2 p.p.m.	4	5	5	5	4–5	5	4
nickel 2 p.p.m.	3–4	5	5	5	4–5	5	4

FADEOMETER HOURS	20	50	100
Top chromed no impurity metals		100%	95%
aluminum 2 p.p.m.		100%	95%
cobalt 2 p.p.m.		100%	100%
copper 2 p.p.m.		90%	85%
iron 2 p.p.m.		95%	90%
nickel 2 p.p.m.		100%	95%

1% Calcochrome Fast Green SGV							
Top chromed no impurity metals	5–	5	5	5	5	5	5
aluminum 2 p.p.m.	5–	5	5	5	5	5	5
cobalt 2 p.p.m.	5–	5	5	5	5	5	5
copper 2 p.p.m.	4–5	5	5	5	5	5	5
iron 2 p.p.m.	4	5	5	5	5	5	5
nickel 2 p.p.m.	5–	5	5	5	5	5	5
POTTING							
Top chromed no impurity metals	4–5	5	5	5	5	5	5
aluminum 2 p.p.m.	4	5	5	5	5	5	5
cobalt 2 p.p.m.	4–5	5	5	5	5	5	5
copper 2 p.p.m.	3	5	5	5	5	5	5
iron 2 p.p.m.	3–4	5	5	5	5	5	5
nickel 2 p.p.m.	4–5	5	5	5	5	5	5

FADEOMETER HOURS	20	50	100
Top chrome no impurity metals		80%	65%
aluminum 2 p.p.m.		75%	64%
cobalt 2 p.p.m.		80%	70%
copper 2 p.p.m.		77%	70%
iron 2 p.p.m.	78%	65%	49%
nickel 2 p.p.m.		80%	60%

been obtained without their use. However, although leveling agents do not prevent metallization of the dye by aluminum, copper, or iron compounds, with few exceptions, they do prevent the concentration of dye in the tips and damaged areas, and thereby reduce or eliminate the contrast in shade between the tip and body regions.

Effect of Metals on Shade and Fastness Properties

Table 34 lists the shades obtained with a number of chrome dyes when applied first as an acid dye, then topped separately with 0.75% of sodium bichromate, ferrous sulfate, copper sulfate, or aluminum sulfate. In each case one dyeing of each dye was not topped, but was used for comparison, to determine the extent of color change caused by the various metals. The figures in the section under "Visual metal sensitivity" give the minimum concentration in parts per million of each metal which will cause a visually perceptible change in shade of the acid dyeing. The notation -1 is used to indicate less than 1 p.p.m. of metal.

In Table 35, the fastness properties of fifteen of the dyes topped with various metals listed in Table 34 are presented, as determined by A.A.T.C.C. Test Method No. 3 for fastness of dyed wool to laundering. Fastness to light also is given for these dyes.

Effect of Trace Metals on Fastness

Reductions in fastness to carbonizing, decatizing, and fulling, caused by partial metallization of dyes due to the presence of iron in top chrome dye baths, has been reported previously *(4)*. Extensive fastness tests were made during this investigation, on top-chromed dyeings produced with additions of 2 p.p.m. of aluminum, cobalt, copper, iron, and nickel to the dye bath at the beginning of the dyeing cycle. The effect of these metals on fastness to laundering, potting, and light was determined by A.A.T.C.C. test methods on each dyeing, followed by comparison against normal dyeings made without the addition of impurity metals. A.A.T.C.C. fastness to laundering test No. 3 for wool was used. Fading was measured on the spectrophotometer after 50 and 100 hours exposure in the fadeometer, and was then compared with unexposed samples. A few dyeings showed a definite break after a short exposure, and these were measured at 20 hours. The results of these tests for several dyes are presented in Table 36.

The author is indebted to Dr. L. H. Turl and Chester A. Amick for assistance in the preparation of the text; to F. C.

TABLE 36—CONTINUED
AATCC Wool Wash Test #3
Staining

	Shade Change	Cotton	Rayon	Silk	Nylon	Acetate	Wool
1% Calcochrome Violet B							
Top chromed no impurity metals	5	5	5	5	5	5	5
aluminum 2 p.p.m.	5	5	5	5	5	5	5
cobalt 2 p.p.m.	5	5	5	5	5	5	5
copper 2 p.p.m.	5–	5	5	4	5	5	4
iron 2 p.p.m.	5	5	5	5	5	5	5–
nickel 2 p.p.m.	5	5	5	4–5	5	5	5–
POTTING							
Top chromed no impurity metals	4–5	5	5	5	5	5	5
aluminum 2 p.p.m.	4	5	5	5	5	5	4–5
cobalt 2 p.p.m.	4	5	5	5	5	5	5–
copper 2 p.p.m.	3	5	5	5	4–5	5	5–
iron 2 p.p.m.	3–4	5	5	5	4–5	5	5–
nickel 2 p.p.m	4	5	5	5	5	5	5–

FADEOMETER HOURS	20	50	100
Top chromed no impurity metals		100%	100%
aluminum 2 p.p.m.		95%	95%
cobalt 2 p.p.m.		98%	98%
copper 2 p.p.m.		94%	94%
iron 2 p.p.m.		90%	80%
nickel 2 p.p.m.		100%	97%

1% Calcochrome Blue Black Conc.							
Top chromed no impurity metals	5	5	5	5	5	5	5
aluminum 2 p.p.m.	5–	5	5	5	5	5	5
cobalt 2 p.p.m.	4–5	5	5	5	5	5	5
copper 2 p.p.m.	4	5	5	5	5	5	5
iron 2 p.p.m.	4–5	5	5	5	5	5	5
nickel 2 p.p.m.	5–	5	5	5	5	5	5
POTTING							
Top chromed no impurity metals	5–	5	5	5	5	5	5
aluminum 2 p.p.m.	4	5	5	5	5	5	5
cobalt 2 p.p.m.	4	5	5	5	5	5	5
copper 2 p.p.m.	3	5	5	5	5	5	5
iron 2 p.p.m.	3–4	5	5	5	5	5	5
nickel 2 p.p.m.	4–5	5	5	5	5	5	5

FADEOMETER HOURS	20	50	100
Top chromed no impurity metals		83%	73%
aluminum 2 p.p.m.		85%	85%
cobalt 2 p.p.m.		87%	85%
copper 2 p.p.m.		85%	70%
iron 2 p.p.m.	82%	75%	60%
nickel 2 p.p.m.		90%	80%

Dexter for the spectrophotometric curves; to Dr. C. M. Judson for the Geiger counter measurements; to Miss Florence Gustafson for preparation of the cross sections, and to the Microscopical laboratory for preparing the Kodachrome slides used in the lecture.

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Meeting Report, New York Section

THE last meeting of the New York Section was held on April 1st at Kohler's Swiss Chalet, Rochelle Park, New Jersey. A dinner preceded the meeting.

Charles W. Dorn, Chairman, announced the appointment of Henry L. Young, Textile Colors Division, Interchemical Corporation, as chairman of the Corporate Membership Committee. Mr. Young succeeds Ernest Hart, Richmond Piece Dye Works, Inc., who has become Vice-Chairman of the National Corporate Membership Committee. Mr. Dorn also announced the appointment of James J. Marshall, American Aniline Products, Inc., as a representative of the Section on the Transportation Committee for the Atlantic City National Convention in October.

Edward W. Rhael, Sandoz Chemical Works, Inc., presented a paper "The Elements of Color and Spectrophotometry". Weldon G. Helmus, Technical Program Chairman, read an anonymously prepared paper on "Jig Dyeing and Advantages of Housings".

It was announced that the May 6th meeting would be the annual business meeting and that an internationally famous speaker would address us on a subject of vital interest to every member.

The attendance was approximately 250.

Respectfully submitted,
NORMAN A. JOHNSON, *Secretary*

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Philadelphia Textile Institute Student Chapter

ON the evening of March 31st, Arthur W. Etchells, Superintendent of Dyeing for the Hellwig Dyeing Corporation, addressed the Philadelphia Textile Institute Student Chapter on "The Dyeing of Rayon and Nylon Piece Goods."

Respectfully submitted,
DORIS McALLISTER, *Secretary.*

EMPLOYMENT REGISTER

This column is open for four insertions per year, per member, without charge. Blanks can be obtained from, and filed with, the Secretary of the Association, Lowell Textile Institute, Lowell, Mass. It is understood that these will be open to inspection by prospective employers, who can obtain further information from the Secretary.

49-6

Education: Three years college chemistry.
Experience: Chemist and Supt. of Dyeing, 25 years practical experience on woolens, rayons, nylons, silk, etc.
Age 50; married; references; will work anywhere. 3-21, 4-4, 4-18, 5-2

49-7

Education: Clemson College; expecting B. S. in Textile Chemistry, June, 1949.
Experience: U. S. Army and three years as bookkeeper.
Age 26; unmarried but with one dependent; South preferred, but North acceptable. 3-21, 4-4, 4-18, 5-2

49-8

Education: R. I. School of Design, graduating in June, 1949.
Experience: One summer, research asst.; editor of college year book; war service.
Age 25; married; references. 4-4, 4-18, 5-2, 5-16

49-9

Education: Clemson College, Textile Chemistry & dyeing, graduating in June, 1949.
Age 22; unmarried, references. 4-4, 4-18, 5-2, 5-16

49-10

Education: Bradford Durfee Technical Institute, dyeing and finishing, graduating in June, 1949.
Experience: Summer in dyeing and finishing plant.
Age 22; married; references. 4-18, 5-2, 5-16, 5-30

TRADE NOTES • NEW PRODUCTS

• Meeting, New York Chapter, LTI Alumni

A dinner meeting of the New York Chapter of the Lowell Textile Institute Alumni Association was held on April 6th at the National Republican Club, New York. Speakers of the evening were Dr. Claudius T. Murchison, president of the Cotton Textile Institute, Inc., and Henry F. Herrmann, president of the American Association of Textile Chemists and Colorists.

A. C. Clifford, president of the Alumni Association, read a letter from Prof. A. Edwin Wells, executive secretary of the Association, commenting on recent activities at the Institute and referring to the plans for the Golden Anniversary Reunion of the Alumni Association to be celebrated at the Statler in Boston on May 13th and at the Institute on May 14th. Louis S. Zisman, past president of the New York Chapter, spoke briefly on building plans for the Institute. A telegram was read from Kenneth R. Fox, president of the Institute, regretting his inability to attend.

Among the "old-timers" present were Walwin Barr, Robert R. Sleeper and George Fuller.

Arrangements for the meeting were handled by Arthur Currier, secretary-treasurer of the Chapter, who discussed plans for a business roster of the alumni in the area. Presiding at the meeting was Norman Johnson, president of the chapter.

Norman A. Johnson, President of the New York chapter of the LTI Alumni Association, who presided at the recent meeting.

• New Low Temperature Method for Wool Shrinkage Control

Development of a new process which makes it possible to apply Lanaset Resin to wool at a low temperature was announced by American Cyanamid Company's Textile Resin Department recently. It is stated that the new process requires absolutely no special high temperature equipment of any kind. All successful previous resin applications on wool called for a cure box to obtain the heat which was essential for curing resin on the fabric. In the new process the ordinary drying equipment is said to be entirely adequate. Thoroughly tested during the past year on a wide range of fabrics by runs at woolen mills, it is claimed that it has been demonstrated that the tensile strength of the fabric is not adversely affected.

The amount of resin required for shrinkage control with the new process is reduced by 30 to 50 per cent. A big advantage of the new process is said to be that it does not dull even the most delicate shades such as baby pink and blue, and the wash fastness of all colors is improved. Clear white can also be obtained.

Hand of the various fabric types is maintained, according to Cyanamid, and excellent results have been shown in plant runs on such diverse materials as blankets, flannels, gabardines, and worsteds.

A pretreatment of the wool before dyeing and resin application has been found to be desirable in about 85 per cent of the cases.

It is stated that practical runs show that the normal equipment of mills, operated at normal speeds, is entirely satisfactory for application of Lanaset finish through the new process. This means that the wool fabric, after it has been passed through the Lanaset Resin bath, can simply be run through the dryer at normal speed.

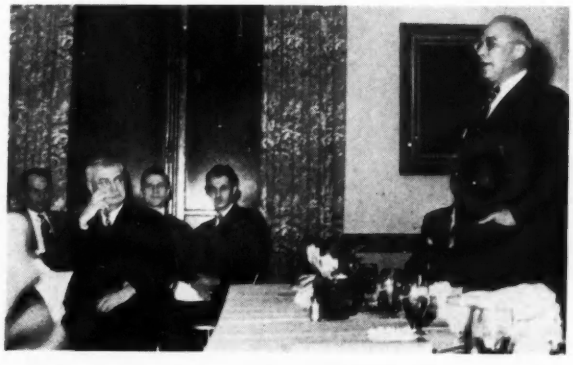
• P.T.I. Corner Stone Ceremony

The ceremony of the laying of the Corner Stone for the new home of the Philadelphia Textile Institute, 3243 School House Lane, Germantown took place at 4 p.m. Thursday, April 21st. Dr. James Creese, President of Drexel Institute, was the speaker on this occasion.

A dinner honoring Dean Richard S. Cox of the Institute was held the evening of Thursday, April 21st, at the Warwick Hotel in Philadelphia. This dinner marked the fiftieth year of association of Richard S. Cox with the Philadelphia Textile Institute. Dean Cox is well known not only to the thousands of Alumni of the School but also to many others in the industry as well.

He has served the Institute in the successive capacities of organizer and head

Henry F. Herrmann, President of the American Association of Textile Chemists and Colorists, who discussed AATCC activities at the meeting of the LTI New York alumni group.



Dr. Claudius T. Murchison, President of the Cotton Textile Institute, who spoke on the future of cotton textiles at the New York chapter meeting of the LTI Alumni Association.

Richard S. Cox

of the jacquard department, dean of the night school for a score of years and as Assistant Director for some twenty years as well. He will continue to serve the school in the capacity of Dean in its new home in Germantown which is to open this fall. Principal speaker for the Cox Commemorative Dinner on April 21st was Alfred H. Williams, President, Federal Reserve Bank of Philadelphia and former Dean of the Wharton School, University of Pennsylvania. Charles W. Carvin of the New York Company of that name served as toastmaster.

A committee comprised of: Leon P. Brick, Howard P. Galloway, Bertrand W. Hayward, Theodore B. Hayward, W. Lyle Holmes, Jr., Rinaldo A. Lukens, F. Everett Nutter, Emil R. Pohlers and Robert A. Smith with Millard D. Brown as Chairman handled the arrangements.

• A-H Moves Boston Office to Providence

Effective April 1, 1949, Arnold, Hoffman & Co. Inc. merged its Boston sales office with the headquarters office in Providence. This consolidation was made in order to better serve its customers. William T. Clarke, former Boston Manager, became the Providence Sales Manager and has supervision over the New England sales force. Mr. Clarke is a graduate of New Bedford Textile School with considerable textile mill and sales experience.

William T. Clarke

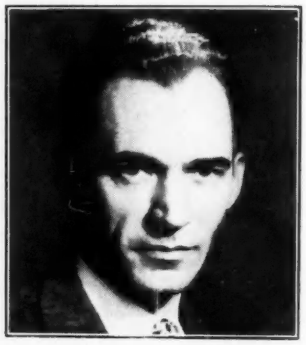
Frank W. Perry

• Stein, Hall Appointments

Stein, Hall & Company, Inc. announces the appointment of Frank W. Perry as manager of the Charlotte, North Carolina office; and Norman H. Nuttall as manager of the Textile Department. Both appointments were made effective March 25th and were announced by Morris S. Rosenthal, President of Stein, Hall.

Frank Perry takes over the supervision of the Charlotte office after a long association with Stein, Hall. A college graduate with a degree in chemistry, Mr. Perry was for many years assistant to the Chief Chemist of the Amoskeag Manufacturing Company, in charge of the general chemical laboratory and associated research and development work. He joined Stein, Hall & Company in July 1937 as a chemist in the Stein, Hall Textile Laboratory, and then became a member of the sales staff. Transferred to the New York office in 1944, Mr. Perry was made manager of the Textile Sales Department the following year.

Norman Nuttall, who moves into the post left vacant by Mr. Perry's appointment as Charlotte sales manager, has been associated with Stein, Hall for nearly ten years. A graduate of the Citadel with a B. S. in chemistry, Mr. Nuttall has had extensive working experience in textiles. Since his association with Stein, Hall in 1940, he has devoted his time principally to laboratory problems connected with the textile industry, but he has also been active for many years in selling.



Norman H. Nuttall

• Warwick Chemical Appointment

Ernest Nathan, president of the Warwick Chemical Co., Division of Sun Chemical Corporation, announces the appointment of Donald J. Eccleston as Sales Manager of their Suntone Pigment Colors for textile printing and dyeing.

Mr. Eccleston comes to Warwick with a wide experience in the sale and application of these materials. He spent five years with the Aridye Corporation as Service Manager, and for the last three years was Assistant General Manager of the Glasgo Finishing Co., Glasgo, Conn.

• Wyandotte Appointment

Thomas H. Vaughn, vice president—research and development, Wyandotte Chemicals Corporation recently announced the appointment of W. L. Rippeteau as manager of development for the company. Mr. Rippeteau joined Wyandotte Chemicals in 1947 as a contact representative for the new organic and inorganic products being produced by the company.

W. L. Rippeteau

After graduation from the University of Kansas in 1939, Mr. Rippeteau was employed by Phillips Petroleum as a research and development engineer. He served during World War II in the Production Engineering Branch of the Technical Division of the office of the Chief of Chemical Warfare Service and was discharged as a Major.

• U. S. Testing Booklets

United States Testing Company, Inc., 1415 Park Avenue, Hoboken, New Jersey, announces the availability of the following booklets:

Testing Price List—lists the various tests made and shows the scope of facilities.

Your Guide to Wise Buying and *Certification*—two pamphlets outlining the Certification Plan and explaining the value of informative labeling.

Core Testing for Determination of Wool Yield and Shrinkage—explains the method for determining the shrinkage

or yield of grease wool, carpet wool or pulled wool.

Testing Terms for Better Understanding—explains the important tests of merchandise, how they are made, what the results mean and minimum specifications.

Testing and Research in Modern Industry—outlines the services of U. S. Testing and contains various tables of equivalents, conversion factors and other useful data.

• Philadelphia Quartz Appointment

The Philadelphia Quartz Company, Philadelphia, Pa., has appointed Richard D. Greenway to its sales staff. He will assume duties in Maryland, Virginia and North Carolina. A native of St. Louis, Missouri, Mr. Greenway was graduated from Missouri University with a bachelor's degree in Chemistry. During World War II he served as an Ensign in the Navy.

Mr. Greenway completed the company's training course in silicate uses to industry and has been covering the eastern Pennsylvania territory for the past five months.

• Hooker Sales Changes

The Hooker Electrochemical Company announces the promotion of S. I. Anderson as assistant to the general sales manager with responsibilities in both eastern and western sales activities. Mr. Anderson, who has been with the Hooker sales department since 1933, has previously been its mid-west representative and mid-west sales supervisor. Prior to his present position he was assistant eastern sales manager.

T. H. Trimble, with Hooker since 1934 as a field salesman in several territories including New York State, Pennsylvania and the southeastern seaboard states, is now eastern sales supervisor. Mr. Trimble will be in charge of field sales activities and the company salesmen in the area from the eastern Ohio boundary thru the Atlantic states. Headquarters for both Anderson and Trimble will be at the Niagara Falls, N. Y. office.

T. H. Trimble

Arthur T. Bennett

Neil M. Barber, who has been with Hooker since 1941, and a sales representative for New York State and western Pennsylvania since 1946, will be assigned to the area comprising eastern Pennsylvania and the southeastern seaboard states.

• Changes in Mathieson Management

Mathieson Chemical Corporation has made several changes in operating management due to the increase in the number of plants resulting from recent acquisitions. Dr. Carl F. Prutton has been appointed vice president-director of operations of all the company's plants and Arthur T. Bennett, vice president, was placed in charge of the eight plants acquired from Southern Acid and Sulphur Company. R. B. Worthy and J. F. Newell were named vice presidents.

Thomas S. Nichols, president of the company, announced that Dr. Prutton, who joined Mathieson last year as director of research, will have charge of research and supervise operations of all the company's plants including those recently acquired. His headquarters will be in the Mathieson New York offices. Mr. Bennett will have his headquarters in Houston. Mr. Worthy will be in charge of the plants acquired from Standard Wholesale Phosphate and Acid Works as well as the Saltville, Va., plant, of which he has been manager. Mr. Newell remains at Lake Charles, La., where he has been in charge of the alkali and ammonia plants.

• Joins Westvaco Sales

James R. Harris, Jr., for the past several years associated with Eagle Picher Company in research and sales capacities, has joined Westvaco Chemical Division, Food Machinery and Chemical Corporation, New York, in the Phosphate Sales Division.

• Southern Research Institute Appointment

Richard E. Hudson, Jr. has been appointed Head of the Textiles Section, Applied Chemistry Division, Southern Research Institute. He was formerly a research and development engineer with West Point Manufacturing Company. Mr. Hudson received his B.S. in Chemical Engineering at Georgia School of Technology. He has had a number of years of experience in textile research work, as well as experience in acid and explosives manufacturing. He has had a number of journal articles published and is a member of the American Association of Textile Chemists and Colorists and the American Chemical Society.

S. I. Anderson

Carl F. Prutton

Richard E. Hudson, Jr.

• New Plant for Textile Colors Division, IC

Construction of a plant in Hawthorne, N. J., to house the operations of its Textile Colors Division, was announced recently by Interchemical Corporation. Work will begin immediately on a group of interconnected buildings, mostly of one-story construction. Plans call for an office and laboratory building, and units for manufacture of the Company's "Aridye" pigment colors for printing textiles and "Interchem" dyes for dyeing and printing acetate rayon, rayon, cotton and other materials. H. K. Ferguson Company, Engineers, of New York, have incorporated in the plans recent advances in structural design, ventilation, lighting and equipment layout.

Operations of the Company, presently conducted at Fair Lawn and at two locations in Paterson, will be transferred, for the most part, to the new location late this year and early in 1950. Established in 1937 as Aridye Corporation, the Textile Colors Division of Interchemical has grown rapidly and today employs about 250 persons.

• American Cyanamid Appointment

Appointment of William M. Brennen to promote sales of textile resins in the New York City area has been announced by American Cyanamid Company's Textile Resin Department. Mr. Brennen will work under the direction of George C. Ramey, Manager of Sales Promotion and Advertising, and will be located at the local offices of the Company in the RCA Building.

Before joining ACC, Mr. Brennen was sales manager of C. M. Deland, woolens, and formerly manager of the coat and suit division of Talon, Inc.

• Blackman and Uhler to Represent Hilton-Davis

Appointment of the Blackman and Uhler Company of Spartanburg, S. C., to represent Hilton-Davis, Cincinnati, in the southern textile field has been announced by Nelson S. Knaggs, vice-president in charge of sales.

At the same time, Mr. Knaggs made public a new series of trade names for its expanding catalog of textile dyestuffs, naphthols, salts and fast bases. "Hidacid" is the trade name selected for the Hilton-Davis group of acid dyes; "Hidaco" for basic dyes; "Hiltonaphthols" for naphthols; "Hiltosal" for salts of fast bases; and "Hiltonil" for the fast bases.

Blackman-Uhler Company, Inc. was organized by William B. Uhler and P. C. Blackman, Jr., and began business in early 1945 for the purpose of distributing dyestuffs and textile auxiliaries in the southeastern area. Their headquarters are located in Spartanburg, South Carolina, at 182 Ezell Street, which provides ample warehouse, office, and laboratory facilities. Their personnel has now been augmented to provide technical representatives serving the printing, dyeing and finishing trade. They also specialize in fugitive tints for the identification of all textile fibers.

• Syntho-Softener B

A substantive, softening agent in the form of a white paste said to possess unusual properties is being marketed by The Hart Products Corporation, 1440 Broadway, New York. Syntho-Softener B is claimed to assure a soft, full-bodied, velvety hand with maximum draping qualities to cotton, rayon, acetate, Bemberg, wool and nylon fabrics. This product, it is stated, preserves the maximum light fastness of goods dyed with such colors as directs, Rapidogens, Indigosols, vats and the like; mixes readily with resins, starches, gums, etc.; and is very inexpensive to use. Syntho-Softener B is claimed to avoid the troubles which usually occur in the use of cation-active softening agents in that it shows no effect on the shade of dyed goods, no yellowing of whites, no rancidity or deterioration on ageing, and no discoloration on pressing. Syntho-Softener B is readily soluble in hot water, forming solutions which are resistant to mild alkalies, acids or hard water.

• National Starch Promotion

National Starch Products Incorporated announces the promotion of Lester Klempner to sales manager, New York division.

A native of Trenton, N. J., Mr. Klempner graduated from the Wharton School of the University of Pennsylvania. Joining National following his graduation, he was assigned to sales in Philadelphia and later was transferred to Baltimore. Following his discharge from the Navy, with the rank of Lieutenant Commander, he was appointed manager of the southeastern division with offices at Atlanta, and later field manager, mid-western division with headquarters at Chicago.

• International Nickel Bulletin

A new technical bulletin on the resistance of high nickel alloys to corrosion by sulfuric acid has been issued by The International Nickel Company, Inc.

While the bulletin is technical in nature, it is written so that it can be readily understood by non-technical as well as technical staffs of industries in which corrosion by this useful but highly corrosive agent is a problem.

Performance of over 30 different nickel-bearing materials in a wide range of services is discussed. Prepared by members of the Company's Corrosion Engineering Section, the bulletin contains 86 tables and 33 graphs and photographs in addition to text matter.

Problems involving such a wide range of operations from the pickling of steel to petroleum refining and textile processing are discussed. Known as Technical Bulletin T-3, it is available without cost through the offices of The International Nickel Company, Inc., 67 Wall Street, New York 5, N. Y.

• Hatch Price List

Hatch Textile Research, 25 East 26th Street, New York 10, announces the availability of its new price list. Herbert H. Hatch, director, states that no general increase in prices has been made although a few minor upward revisions seemed necessary.

During the past year improved facilities and special equipment have been added.

• Stronach Retires

I. N. Stronach, for the past 30 years employed as General Superintendent of the combined Hampton Company and American Thread Co., Easthampton, Mass., has resigned his position and will retire. Charles W. Taylor for the past 23 years employed by the same companies as Assistant Superintendent will succeed Mr Stronach as General Superintendent.

• Sodium Trifluoroacetate

Pilot plant production of sodium trifluoroacetate is being carried out by the Hooker Electrochemical Company, Niagara Falls, New York. The compound is in the form of a white, odorless, noncorrosive, free flowing powder. Its properties suggest the possibility of its application in the preparation of many useful chemicals such as herbicides, insecticides and dyestuffs. The salt is nontoxic and is 98 to 99% pure. Sodium trifluoroacetate is hygroscopic. When converted to the acid, as generally done for chemical processing, the acid is highly ionized. Technical Data Sheet No. 377, describing the salt, is available upon request.

• AIL Representative

The American Institute of Laundering has announced the appointment of John W. Sallis as a special representative for its Certified Washable Seal Program. He will do liaison work between the Institute's national headquarters and laboratories at Joliet, Illinois, and its Textile Approval Division office in New York City.

Mr. Sallis formerly was manager of the Institute's Membership Service Department in Joliet.

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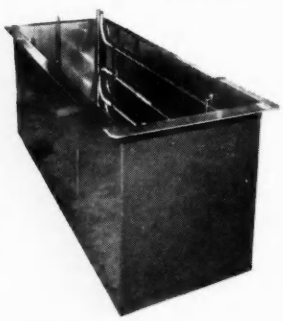

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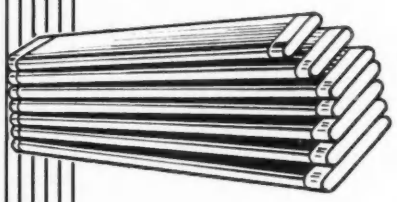

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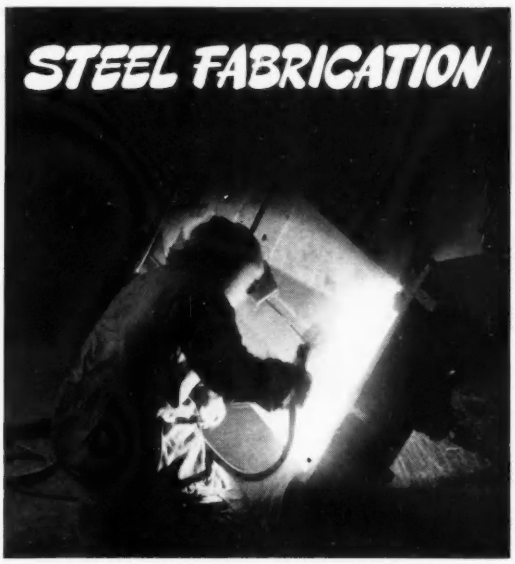

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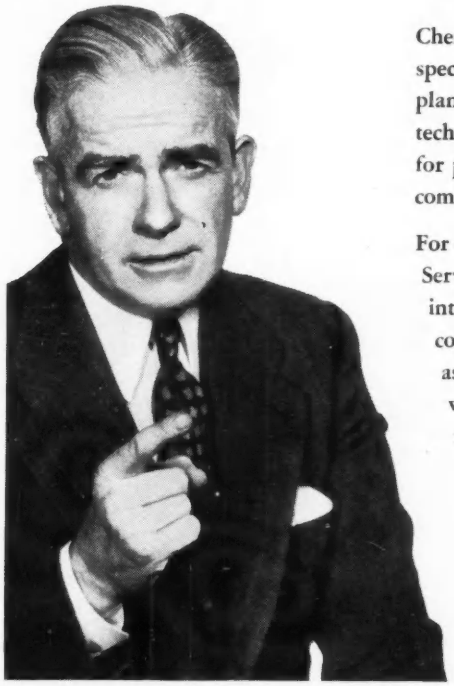

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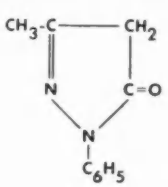

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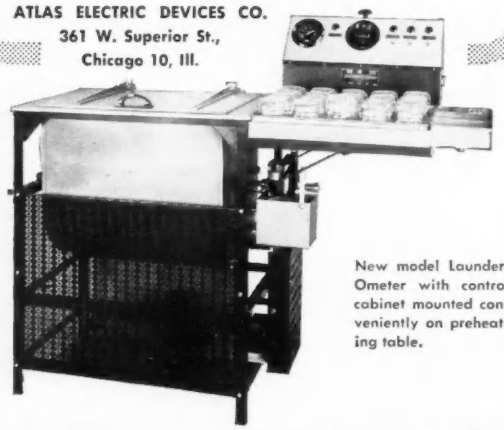

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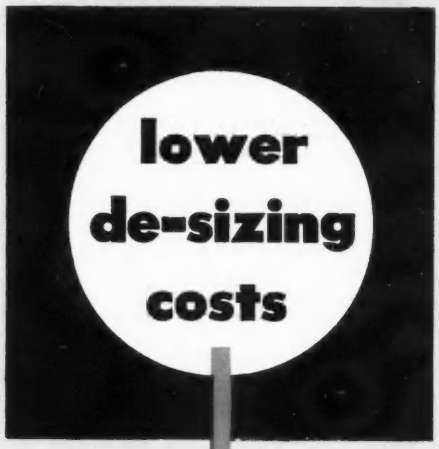

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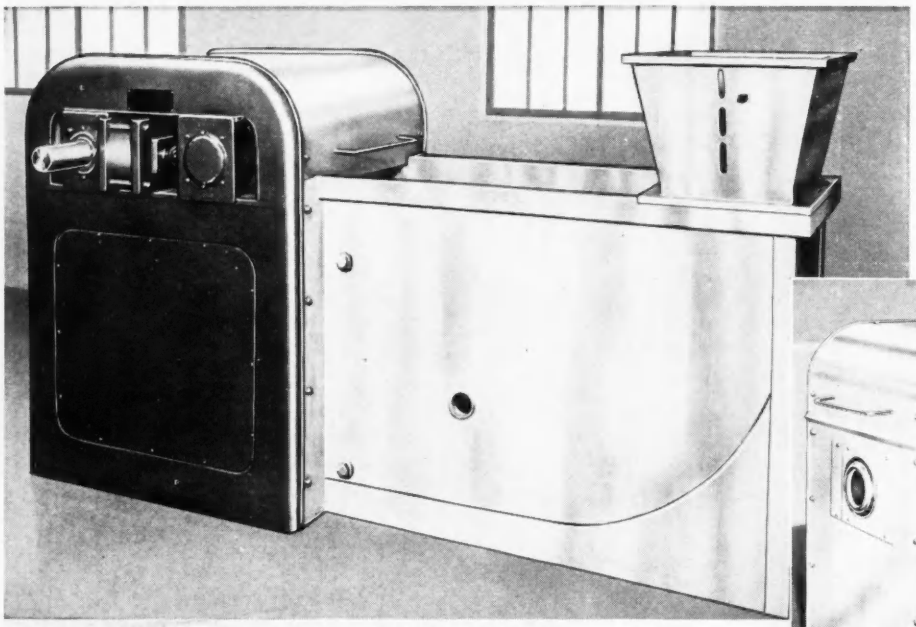

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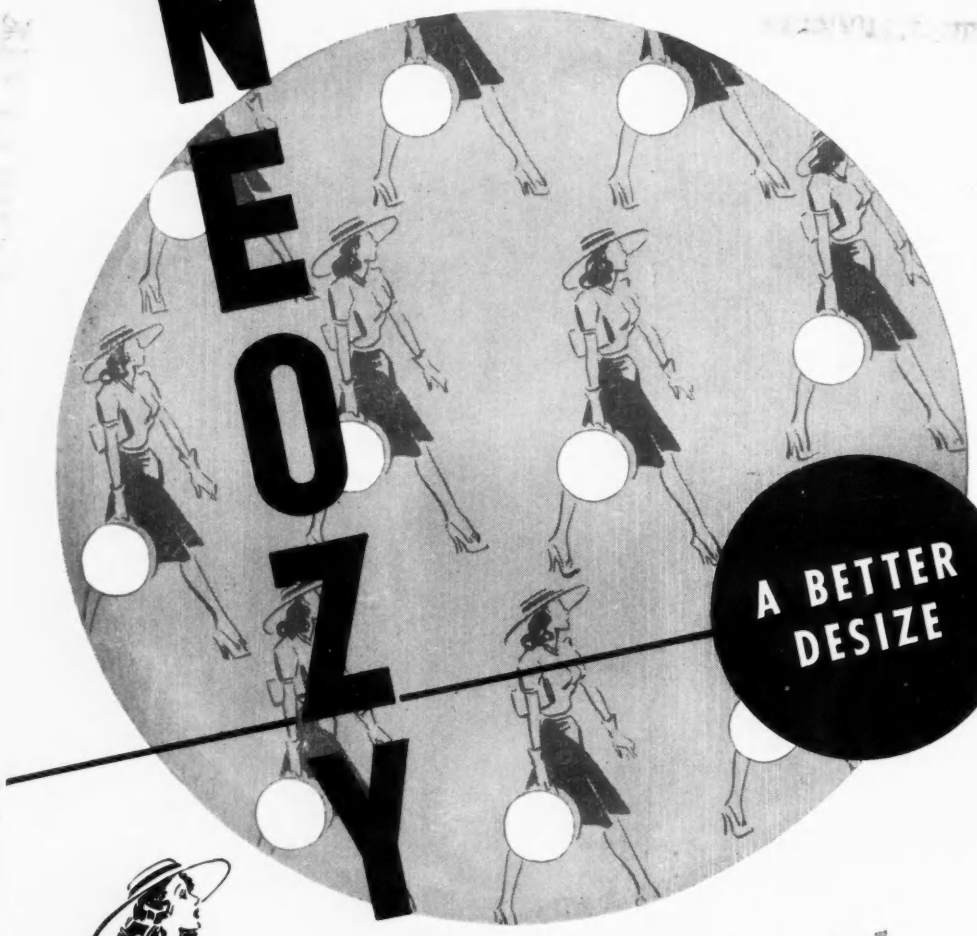

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